SEWAGE AND INDUSTRIAL WASTES

(Formerly Sewage Works Journal)

JOURNAL of the FEDERATION OF SEWAGE AND INDUSTRIAL WASTES ASSOCIATIONS

Special Features

Toxicity Bio-Assay Methods—FSIWA Research Committee

Algae Symbiosis in Oxidation Ponds—Ludwig, et al.

Trickling Filter Media—Schroepfer

Chemical Oxygen Consumed Tests—Riehl

NOVEMBER, 1951—VOL. 23, No. 11

Make Plans to Attend

SILVER ANNIVERSARY MEETING

OF THE

FEDERATION OF SEWAGE AND INDUSTRIAL WASTES ASSOCIATIONS

IN CONJUNCTION WITH

NEW YORK SEWAGE AND INDUSTRIAL WASTES ASSOCIATION

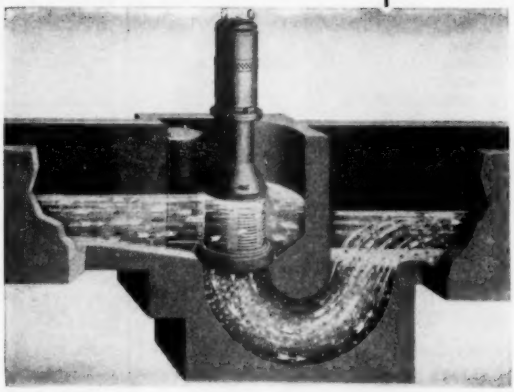
HOTEL STATLER
NEW YORK CITY

October 6-9, 1952

FEDERATION OF SEWAGE AND INDUSTRIAL WASTES ASSOCIATIONS
325 ILLINOIS BUILDING CHAMPAIGN, ILLINOIS

CHICAGO COMMINUTORS

cut coarse sewage matter into small settleable solids without removal from channel



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New York Sewage and Industrial Wastes Association	Hotel Statler, New York, N. Y.	Jan. 17–18, 1952
Texas Water and Sewage Works Association	Texas A. & M., College Station, Texas	March 9–13, 1952
Arizona Sewage and Water Works Association	Maricopa Inn, Mesa, Ariz.	April 3–5, 1952
Arkansas Water and Sewage Conference	Engineering Building, University of Arkansas, Fayetteville, Ark.	April 7–9, 1952
Montana Sewage and Industrial Wastes Association	Northern Hotel, Billings, Mont.	April 10, 1952
Virginia Industrial Wastes and Sewage Works Association	Hotel Roanoke, Roanoke, Va.	May 12–13, 1952
Pennsylvania Sewage and Industrial Wastes Association	Pennsylvania State College, State College, Pa.	Aug. 27–29, 1952

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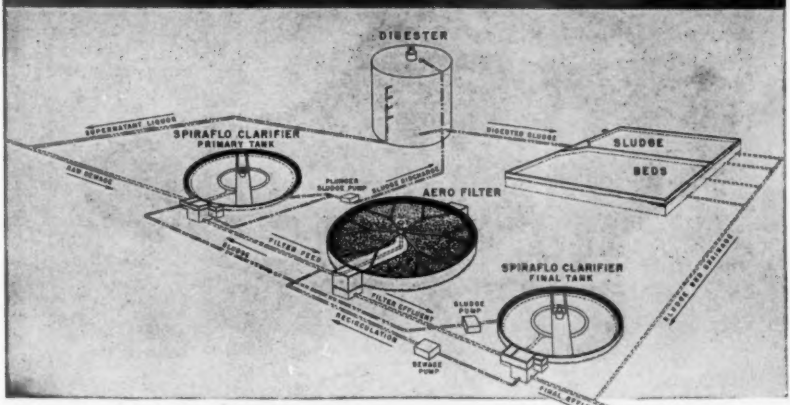

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		Sus. Solids PPM	B.O.D. PPM	Sus. Solids PPM	Per Cent Reduction	B.O.D. PPM	Per Cent Reduction	Sus. Solids PPM	Per Cent Reduction	B.O.D. PPM	Per Cent Reduction	Sus. Solids Per Cent	B.O.D. Per Cent
6-15	0.2996	210	255	79	62.4	136	46.6	28	64.6	18	86.8	86.6	93.0
6-16	0.2467	425	260	85	80.0	127	51.2	23	73.0	19	85.1	94.6	92.6
6-17	0.2554	410	235	124	69.7	141	40.0	28	77.4	18	87.3	93.3	92.4
6-18	0.2487	308	222	76	75.4	108	51.4	34	55.3	16	85.2	89.0	92.8
6-19	0.1753	222	218	64	71.2	132	39.5	17	73.5	—	—	92.4	—
6-20	0.2666	262	320	88	66.4	147	54.1	20	77.3	30	79.6	92.4	90.6
6-21	0.2279	240	215	78	67.5	138	35.8	19	75.7	20	85.7	92.0	90.7
Average	0.2456	297	247	85	70.5	132	45.5	24	71.0	20	85.0	91.5	92.0

* Name of plant furnished on request, as well as operating data from other installations.

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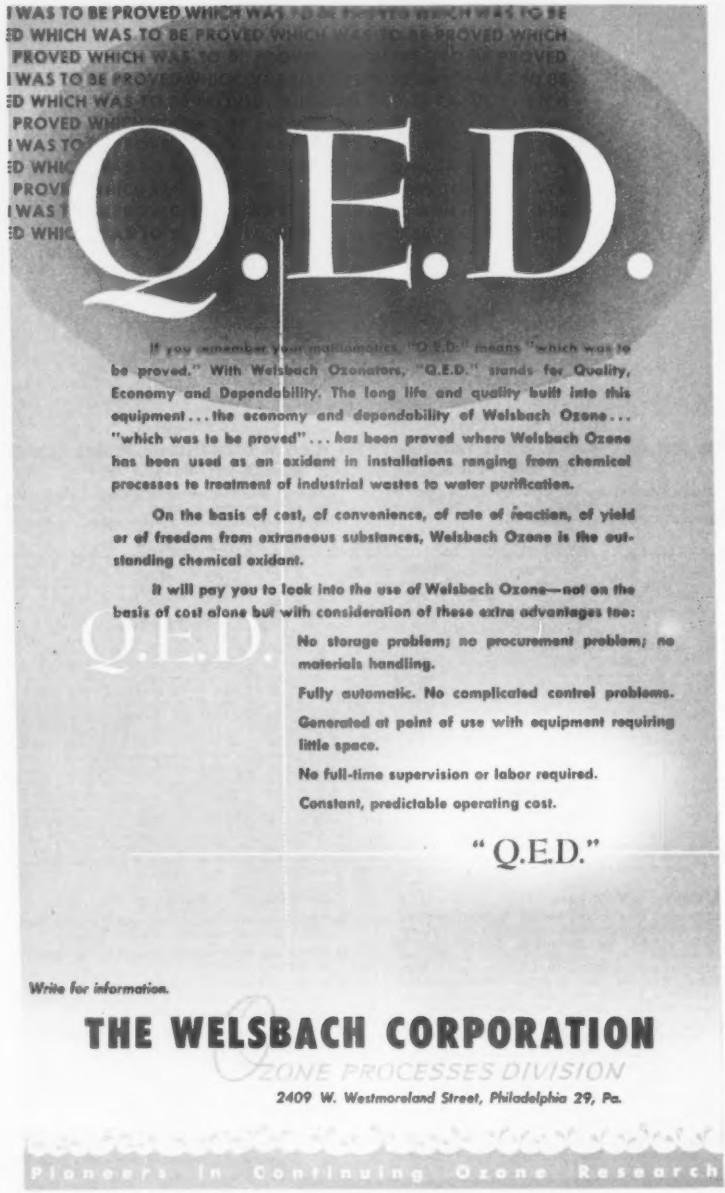
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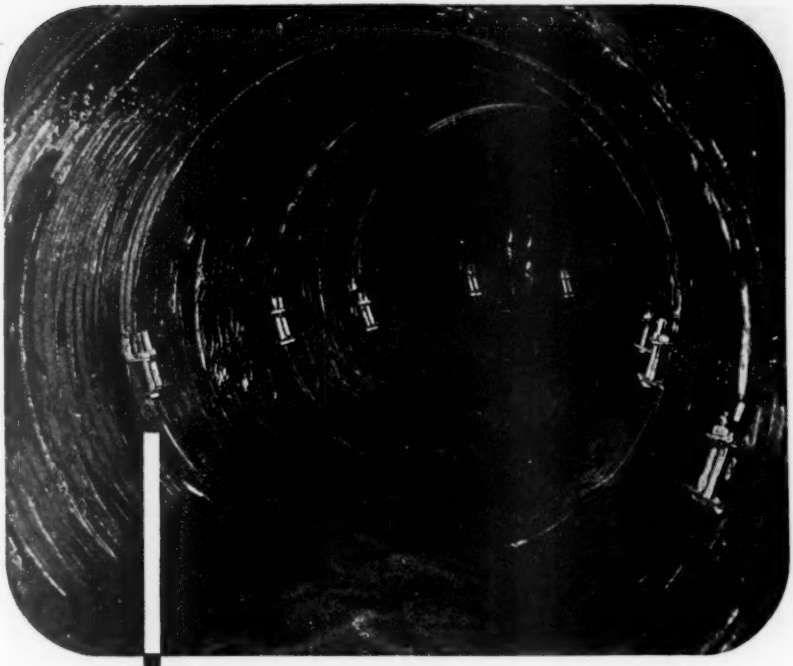

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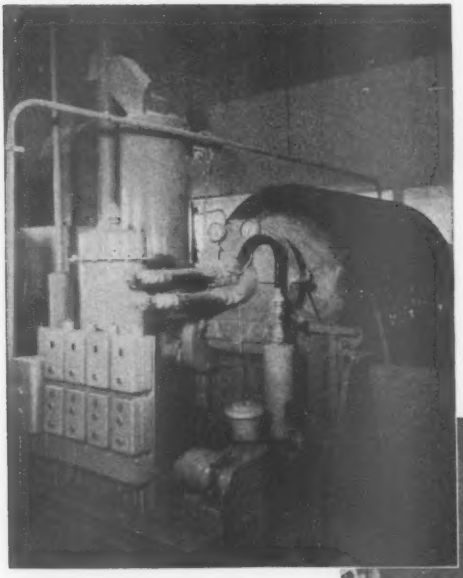

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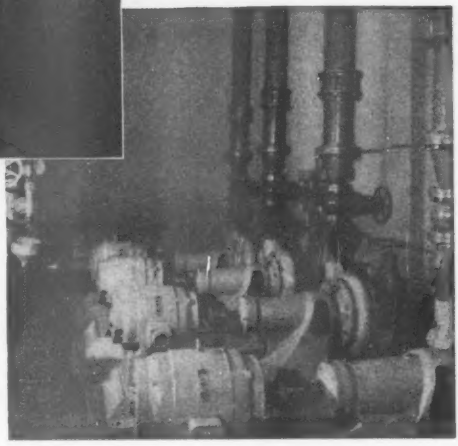

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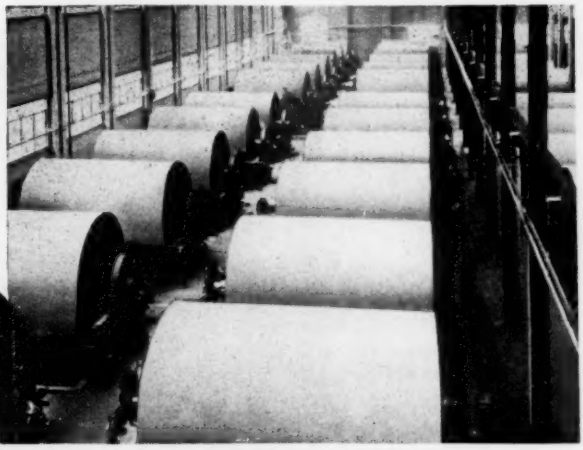

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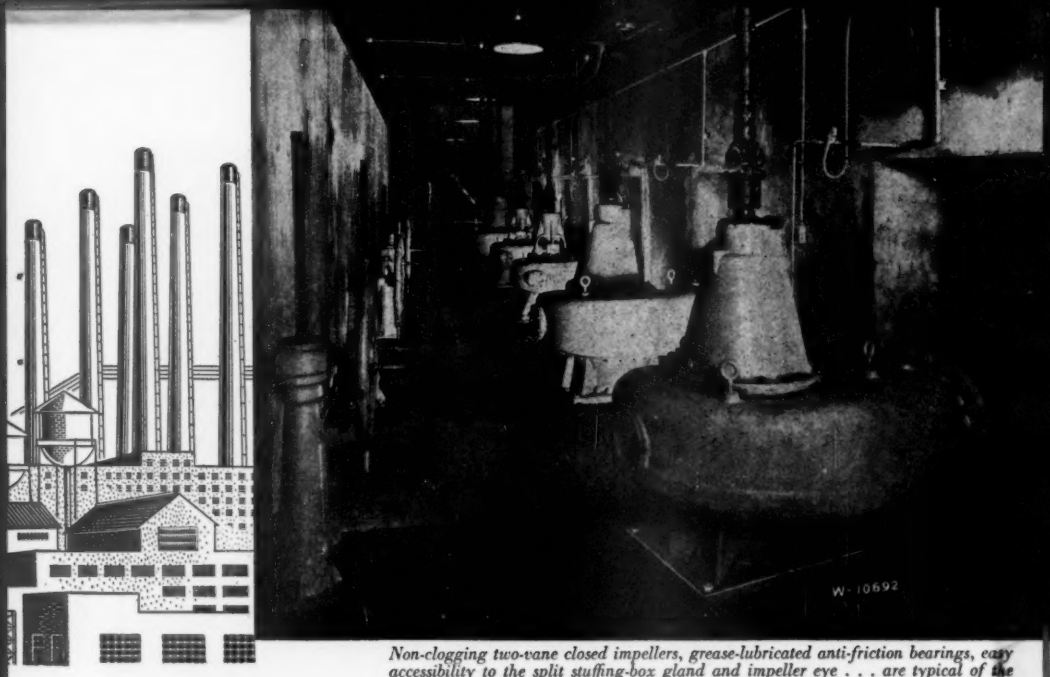

Non-clogging two-vane closed impellers, grease-lubricated anti-friction bearings, easy accessibility to the split stuffing-box gland and impeller eye . . . are typical of the many Worthington MIXFLO *features assuring low-cost, trouble-free sewage pumping.*

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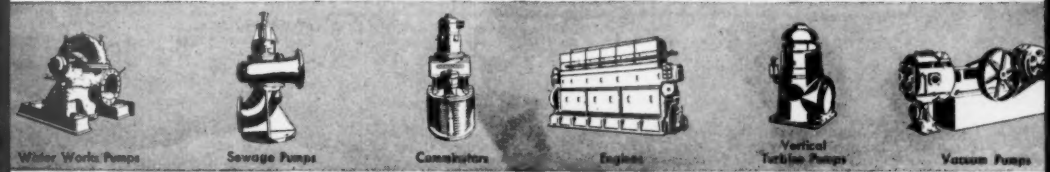

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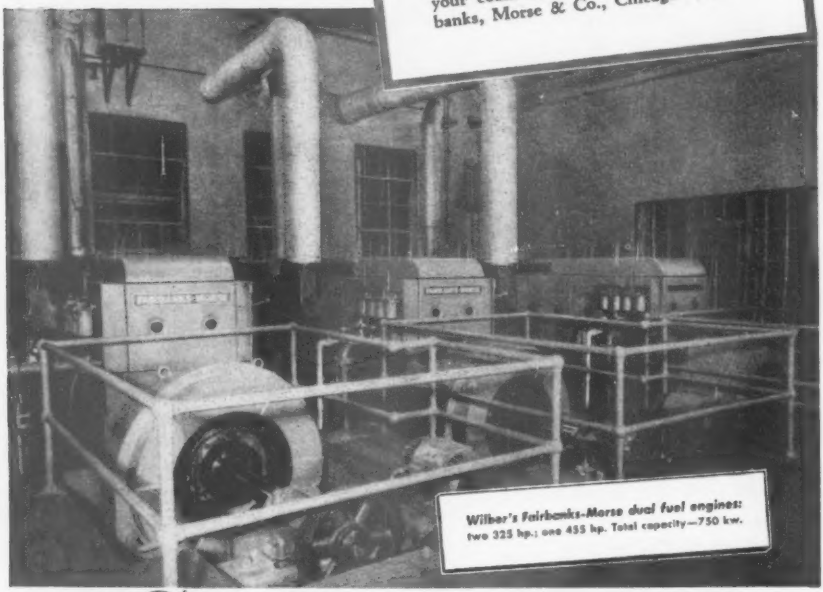

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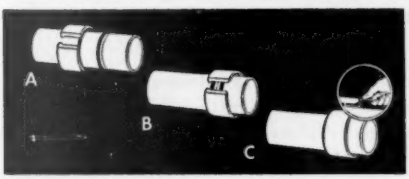

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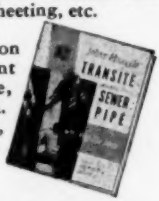

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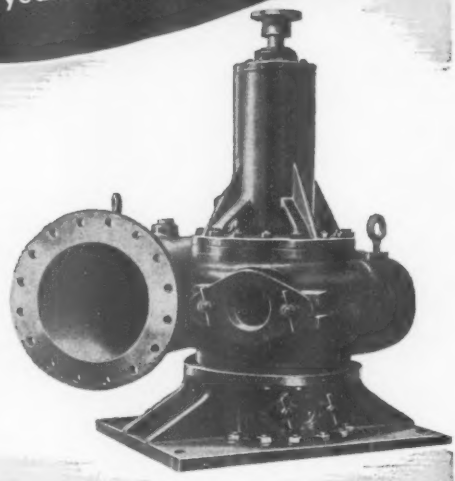

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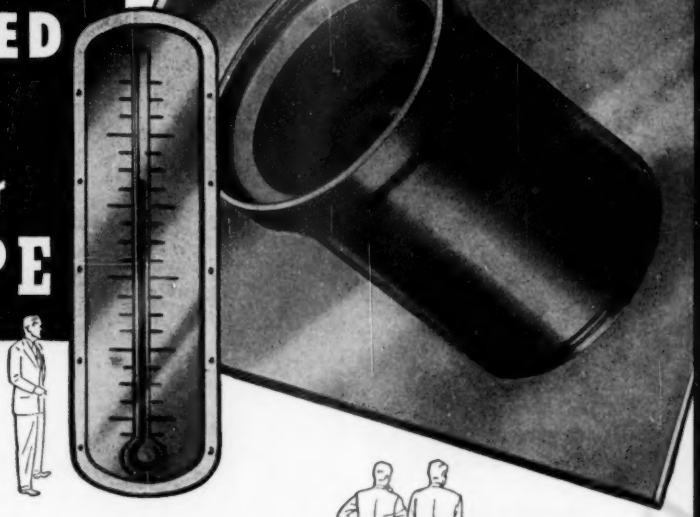

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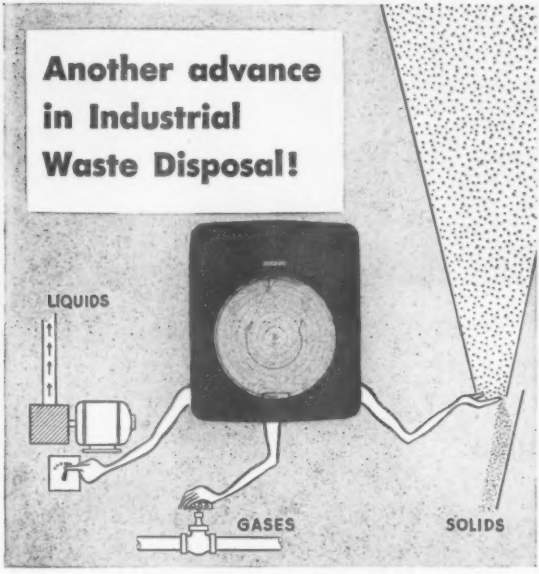

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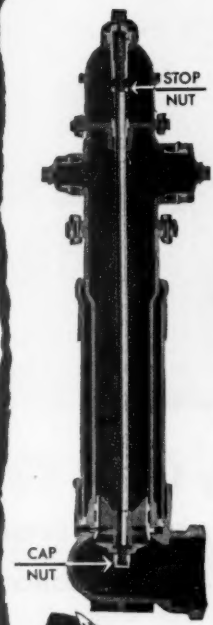

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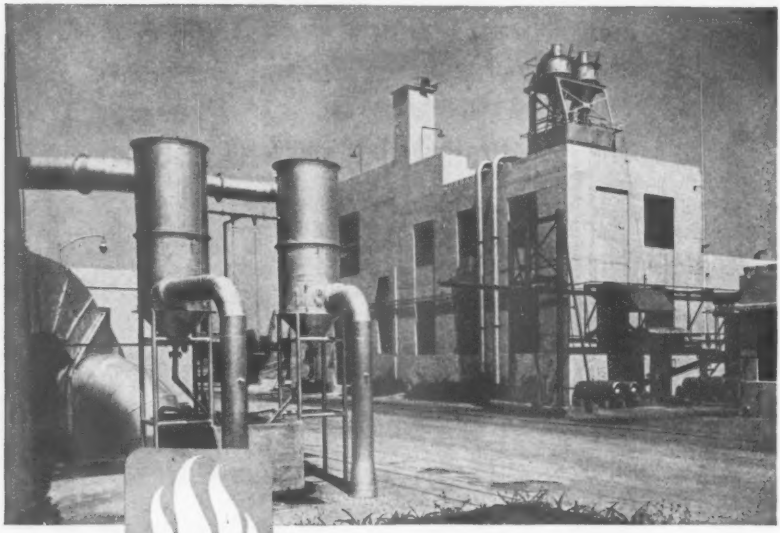

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...almost triples capacity of original installation

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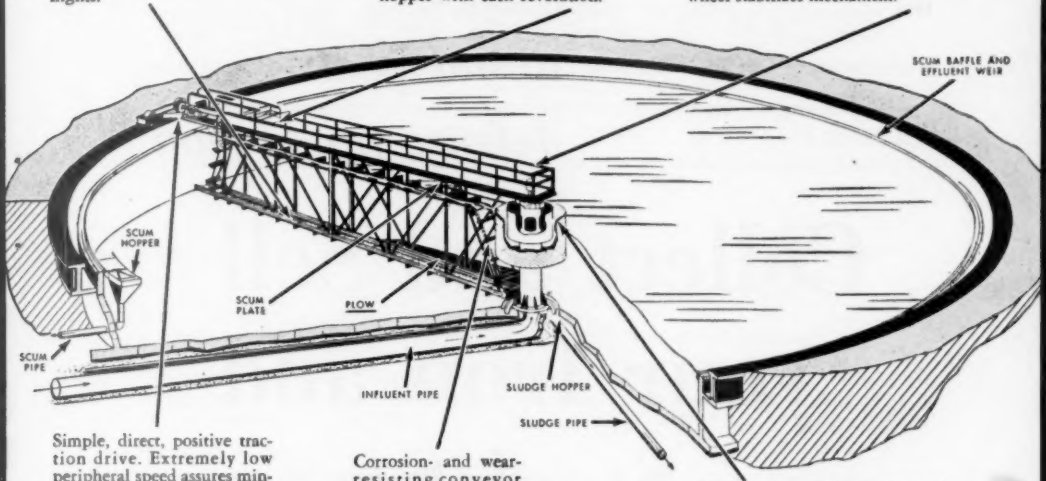

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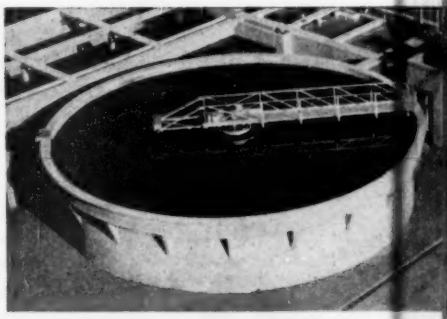

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SEWAGE AND INDUSTRIAL WASTES

Journal of the Federation of Sewage and Industrial Wastes Associations

Volume 23 NOVEMBER, 1951 Number 11

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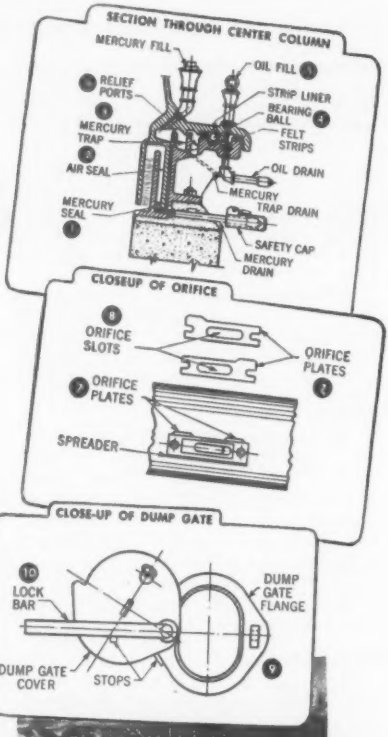

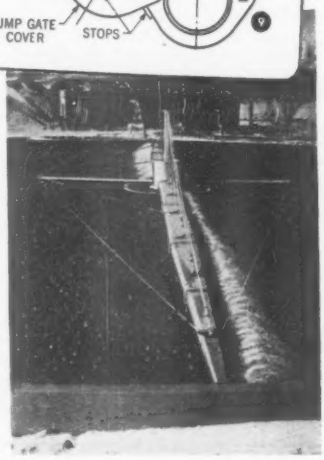

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Sewage Works

ALGAE SYMBIOSIS IN OXIDATION PONDS

I. Growth Characteristics of *Euglena Gracilis* Cultured in Sewage *

By Harvey F. Ludwig, William J. Oswald, H. B. Gotaas, and Victoria Lynch

Respectively, Associate Professor, Research Engineer, Professor, and Research Microbiologist, Sanitary Engineering Laboratories, University of California, Berkeley, Calif.

Introduction

In regions where the climate is sunny and dry, oxidation ponds afford a simple and economical means for biological stabilization of settled sewage and industrial wastes. The pond is usually an artificially constructed shallow basin from about 3 to 5 ft. in depth, with sufficient capacity to provide a flow-through or retention period of from 20 to 30 days. Oxygen is absorbed from the atmosphere, and bacterial oxidation processes convert to carbon dioxide much of the organic carbon initially present in the sewage.

These conditions create an environment suitable for the development of photosynthetic plankton or algae. The presence of carbon dioxide, nitrogen in various forms, phosphorus, and other trace elements, is favorable to the growth of algae; and these, through photosynthesis, produce an excess of oxygen above their own respiratory requirements. The algae are particularly numerous in the outer sections of the pond, where, during the daylight hours, the water may become supersaturated with oxygen. In some installations a portion of the pond effluent is recirculated to the influent end. This transfers some of the surplus oxygen to where it is most critically needed to maintain an aerobic environment and also, through seeding, promotes the growth of algae in the incoming sewage.

The treatment processes occurring in the pond may therefore be said to result from a symbiosis of bacteria and algae. However, the role of the algae has not been precisely established, so that it is not certain to what extent the algae are essential to proper pond functioning. To investigate this role, the authors have undertaken laboratory-scale studies involving the culture in sewage of a single alga, *Euglena gracilis*, a species commonly found in oxidation ponds and in polluted waters. Measurements have been made to ascertain the growth characteristics of this species and also its effects upon the added sewage.

The studies to date have been conducted in specially-designed culture tubes (Figure 1), described in more detail in an earlier report (1). A definite volume of culture is removed each day, depending upon the retention period being studied, and is replaced by an equal volume of settled sewage. The portions of settled sewage employed each day were all derived from a single 100-gal. sample, which had been previously settled, pasteurized (to minimize changes prior to

* Presented at meeting of Ecological Society of America; Los Angeles, Calif.; June 20, 1951.

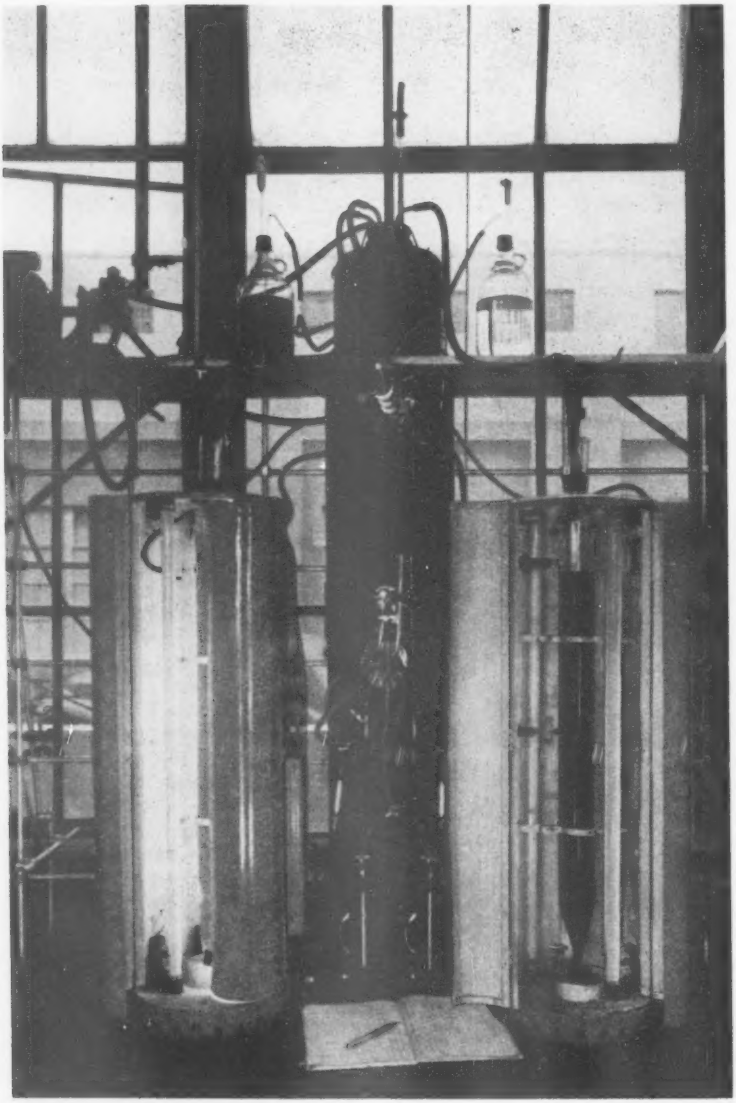

FIGURE 1.—Algae culture tubes.

sterilization), bottled in half-gallon lots, sterilized, stored at low temperature, and reseeded with sewage bacteria just prior to use, as described elsewhere (1). The processed sewage had a pH of 6.7 and other characteristics as follows: alkalinity, 50 p.p.m.; manometric B.O.D. (5-day, 25° C.), 112 p.p.m.;

suspended solids, 22 p.p.m. (including 20 p.p.m. volatile suspended solids); total solids, 620 p.p.m.; total ash, 380 p.p.m.; total volatile solids, 240; organic carbon, 73; organic hydrogen, 7.3; Ca, 35; Mg, 5; K, 10 p.p.m.; Mn, 0.2 p.p.m.; NH_4, 32 p.p.m.; and PO_4, 25 p.p.m. The 100-gal. sample was obtained from the San Francisco Richmond-Sunset sewage treatment plant, which receives essentially domestic sewage.

The range of retention periods investigated has been varied from 2 to 20 days, and correspondingly the environment to which the algae have been exposed varies progressively from very favorable (abundant nutrients) to very unfavorable (shortage of one or more nutrients). Constant mixing is maintained, in this case by bubbling air at the rate of 500 ml. per minute up through the column of liquid. The present work, designated the Series III studies, employed ordinary air for bubbling, without any added CO_2. The earlier studies (1), designated Series I and II, used air enriched with 2.30 and 1.75 per cent CO_2, by volume, respectively, and were made with a sewage having a higher alkalinity (200 p.p.m.).

The culture is continuously exposed to fluorescent light, held constant and averaging about 1,200 foot-candles within the tube. The temperature of the culture is maintained constant at 25° C. by a heat-exchanging pipe placed within the culture. Each test is begun by filling the tube with a previously prepared culture, grown in the same sewage, which has an initial cell count of 0.25×10^6 cells per ml. With this procedure a minimum time period is required to bring the system to a condition of equilibrium. Care is exercised during progress of the test to prevent biological contamination.

Due to the abundant and continuous lighting, the activity of the algae in the tubes is accelerated with respect to what occurs under field conditions. The average *Euglena* population present in the tubes at equilibrium is of the order of 2 to 10 times the population of all types of algae usually present in pond effluents. The results reported here, therefore, represent a type of "accelerated symbiosis" in which the relative roles of algae and bacteria are not strictly comparable to conditions in actual ponds. Furthermore, the air bubbling previously mentioned makes possible a rate of exchange of oxygen and carbon dioxide different from that obtained in the field, where the only exchange is that with the atmosphere at the pond surface. Some recent studies (to be published later) involve use of a closed system, wherein mixing is accomplished by recirculation and bubbling is not used. The data obtained will permit calculation of a precise materials balance between the influent nutrient and effluent culture, taking account of changes in the gas plenum above the liquid surface.

Characteristics of *Euglena* Cells

Several criteria have been developed for evaluating the characteristic properties of the *Euglena* cells found in the culture at condition of equilibrium. These characteristics include the following:

1. Cell count, C. The number or population of cells per ml. of culture at equilibrium. A hyperbolic curve is obtained when cell counts are plotted as ordinates against elapsed times as abscissas, following the initial filling of the tube. Equilibrium is attained when the cell count, which is increasing from its initial seed value, levels off at or near its maximum value. Equilibrium is usually reached after a period equal to one retention period plus 2 or 3 days.

2. Cell shape. The average ratio of length to width of individual cells.

3. Cell volume, V_m. The real volume of a million cells, in cu. mm., as de-

termined by direct microscopic measurement of some of the cells.

4. Packed volume, V_c. The centrifuged volume, in ml. of the cells contained in 1 l. of culture, after centrifuging at 500 *g*. The result is a function of the cell count, the cell volume, and the cell shape.

5. Cell weight. The dry weight of the cells in μg. (10^{-6}g.) per million cells.

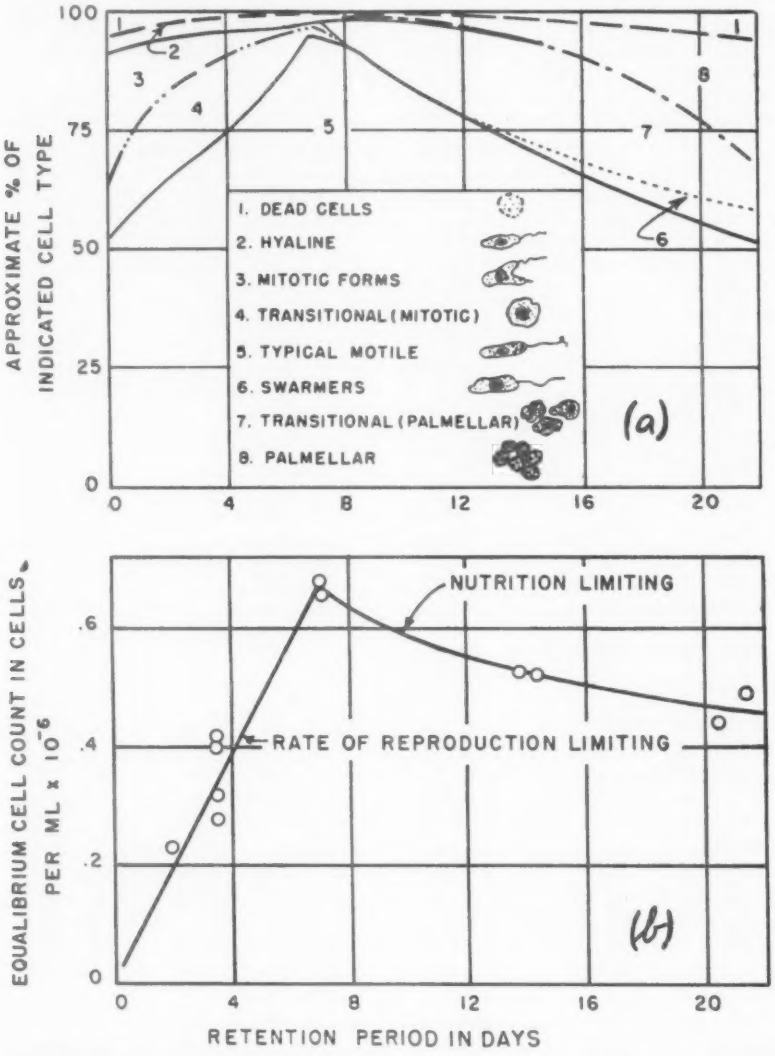

FIGURE 2.—Number and distribution by types of *E. gracilis* cells grown at varying retention periods.

6. Cell density. The dry weight of the cells in mg. per cu. mm. of real volume.

7. Cell index, *I*. The packed volume per million cells; that is, V_c/C. The cell index numerically equals the packed volume in cu. mm. of a million cells.

8. Cell color. This varies from dark green to pea green to yellow-green to yellow to yellow-brown, depending upon the cell types and availability of nutrients.

9. Cell types. Various types or forms of *Euglena* may be distinguished, depending on the "age" of the individual cells (see Figure 2), as follows:

1. Motile forms—Typical *E. gracilis* (type commonly illustrated in texts).
2. Hyaline forms—Forms without visible chlorophyll.
3. Mitotic forms—Forms undergoing binary fission.
4. Swarmers—Motile forms "hatching" from palmellar forms.
5. Palmellar forms—Forms at resting stage.
6. Transitional forms—Forms intermediate between motile or mitotic forms, or between motile and palmellar forms.

Two other culture characteristics, the cell concentration, *D*, and the yield of the culture, *Y*, are discussed in a later section.

Cell Types and Cell Counts

Figure 2a summarizes the observations of cell types, as a function of retention period, and also shows sketches of each type. Figure 2b is a plot of cell counts at equilibrium.

The "most vigorous" condition is seen to occur when the retention period approximates seven days; that is, when $R = 7$. At this condition the cell count is at maximum, and practically all cells are active motile types. With shorter retention periods the cells are growing and undergoing division as rapidly as possible, due to the abundance of available nutrients. (The Series I studies (1) showed the generation time for *E. gracilis* to be of the magnitude of 24 hr.) However, the nutrients are removed from the tube before a high population can be built up. With longer retention periods there is a shortage of one or more nutrients, so that cell counts become progressively lower. The motile forms grow more slowly, become larger, and contain more fat. Moreover, with increasing retention periods, the inhibiting condition becomes important enough to change the type of some of the cells, forcing them into palmellar forms of low metabolic activity. Further inhibition can change the palmellar to the swarming form, which appears to be a kind of final vigorous attempt to migrate to a more favorable environment. The highest cell count is evidently fixed by the intersection of two curves, one representing growth limited principally by reproduction rates, and the other limited by nutrients.

In the portions of oxidation ponds where *Euglena* are present, their types and counts depend upon the "effective retention period"—that is, the environment actually obtaining—which is determined by the available nutrients and light. The effective retention period is not the same as the hydraulic retention period of the pond, because the *Euglena* are not uniformly distributed throughout the pond, but are concentrated in zones wherein favorable conditions exist.

Actual observations of the types of *Euglena* found in pond samples (1) (2) show them to be principally the motile forms. Along the fringes of favorable zones some of the cells might become converted to the palmellar forms or to swarmers. Palmellar types would tend to sink to the bottom, and therefore not be collected in samples.

Swarmers would probably find their way to favorable zones or else convert to the palmellar stage or die.

Cell Volumes, Shapes, and Colors

Plots of the volume of algal cells contained in 1 l. of culture are shown in Figure 3, employing two different criteria for volume: (a) The actual or real volume, in ml. equal to $C\ V_m\ 10^6$; and (b) the packed volume, V_c, in ml.

It is noted that the real volume curve follows the general shape of the cell count curve; that is, the greater the number of cells present the greater their total volume, despite variations in individual cell size. Figure 4 illustrates the individual variations, and shows the size of the cells to be smallest at approximately the same condition ($R = 7$) associated with maximum cell count.

Comparison of the two curves of Figure 3 shows the packed volume to be greater than the real volume, presumably by the space taken up by the interstices in the packing. Where the cell forms are mostly the motile type, the packing is evidently dense; but where mixtures of different shapes occur the packing becomes loose.

The dissimilarity in the shapes of the different cell forms is indicated by differences in their length-width ratios. Numerous measurements of the average length-width ratio, for all cells in samples corresponding to different retention periods, show a maximum value of 4:1 at $R = 7$, when practically all the cells are of the typical elongated type. The ratio decreases with increasing retention periods, from 4:1 at $R = 7$ to approximately 2.5:1 at $R = 20$, due to the increasing concentration of the more rounded palmellar types. The ratio remains at about 4:1 for $R < 7$, due to the fact that the younger motile forms are more elongated than the older motile forms, which offsets the effect on the average ratio of the more rounded transitional and mitotic forms.

The color of the cells, indicated in Figure 3, changes progressively from dark green to yellow, with the degree of yellow being greater at higher retention periods. This agrees with results previously obtained wherein the bubbling gases were rich in CO_2; except

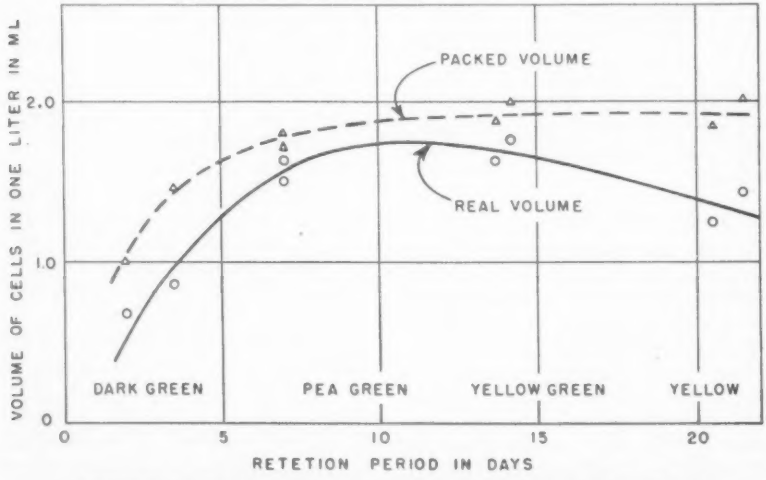

FIGURE 3.—Volume of *E. gracilis* produced per liter of tube effluent.

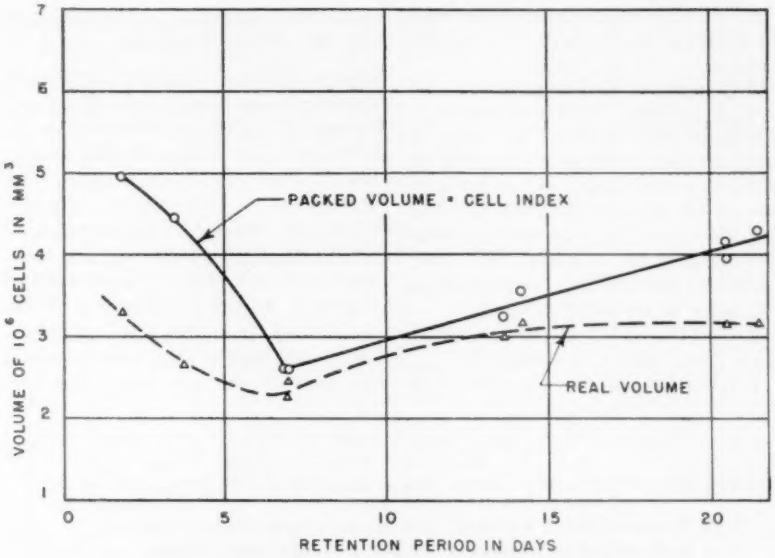

FIGURE 4.—Volume of *E. gracilis* cells per million cells.

that with the greater availability of CO_2 the progression of color, over the same range of retention periods, extended over a wider color range, from very dark green to yellow to brown.

Cell Weight and Density

A plot (not shown) of cell weight versus retention time results in a smooth curve, with a definite minimum point. At $R = 2$ the weight is 0.9 μg./10^6 cells, and with increasing retention period this decreases to the minimum value, 0.56 μg./10^6 cells, at $R = 7$. It then increases with increasing retention period throughout the range tested, to a maximum of 1.22 μg./10^6 cells at $R = 20$.

The minimum cell weight is thus associated with the same environment ($R = 7$) as the minimum cell volume, maximum cell count, and minimum cell index.

By dividing the values for cell weight by those for real cell volume, data are obtained which express the density of the individual cells. Such calculations show that the older cells are considerably more dense. Expressed in milligrams dry weight per cubic millimeter of real volume, the cell density lies between 0.25 and 0.30 for $R < 14$. Beyond $R = 14$ the density increases progressively to 0.40 at $R = 20$. This increase in density reflects a change in composition of the cells; specifically, a decrease in water content of the cells with increase in fat content. Similar findings with *Chlorella*, made by Spoehr and Milnar, are reported by Smith (3).

Oxygen Balance

The capacity of the culture, and of each of its constituents, for absorbing or releasing oxygen was studied using the Warburg manometric method, employing the techniques described in an earlier publication (4). For each sample the following oxygen exchange curves were obtained (at a temperature of 25° C.):

1. B.O.D. of whole culture, with algae living, in the dark (foot-candles = 0).

2. Oxygen released by whole culture, with algae living, with foot-candles = 250 inside the test flask. This reduced light intensity, compared with that inside the culture tube, causes a reduction in the rate of photosynthesis (5) (6); however, the rate of photosynthesis inside the Warburg flask corresponds more closely to conditions obtaining in receiving streams.

3. B.O.D. of whole culture, with algae dead (killed by freezing).

4. B.O.D. of algae-free culture; that is, of the supernatant after centrifuging the sample of culture.

Figure 5 shows a typical set of such curves for one sample, and also a plot (Curve E) showing the B.O.D. of the added sewage. From the data of Figure 5, other "corrected" curves (not shown) were prepared to represent (Curve F) the total oxygen produced by photosynthesis—that is the ordinates of Curve B plus the ordinates of Curve A, or the total oxygen photosynthetized, including that simultaneously used up in algal and bacterial respiraton—and the respiration re-

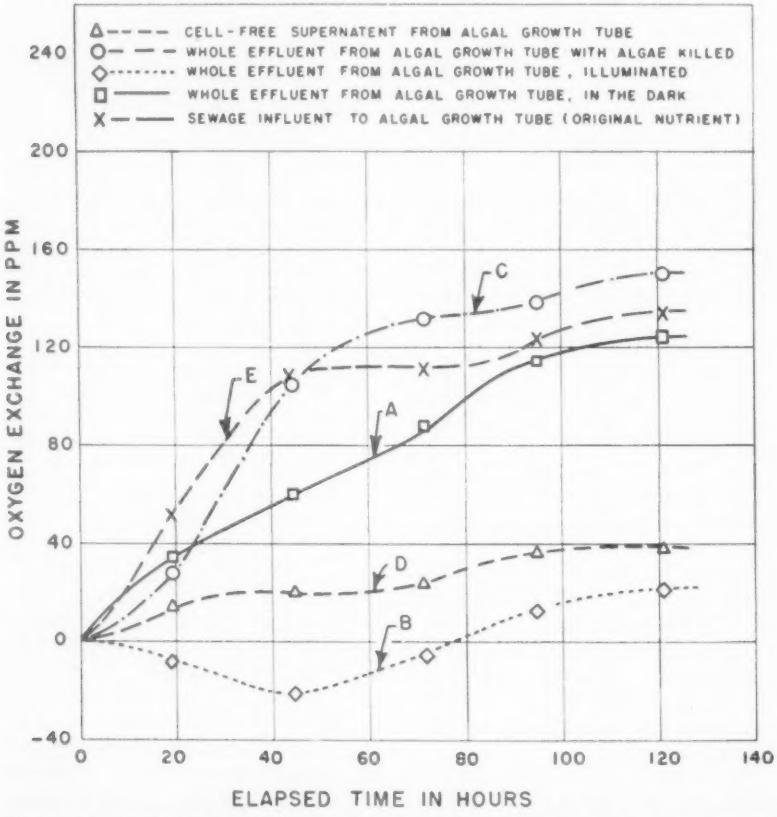

FIGURE 5.—Typical manometric oxygen uptake and production curves for culture sample.

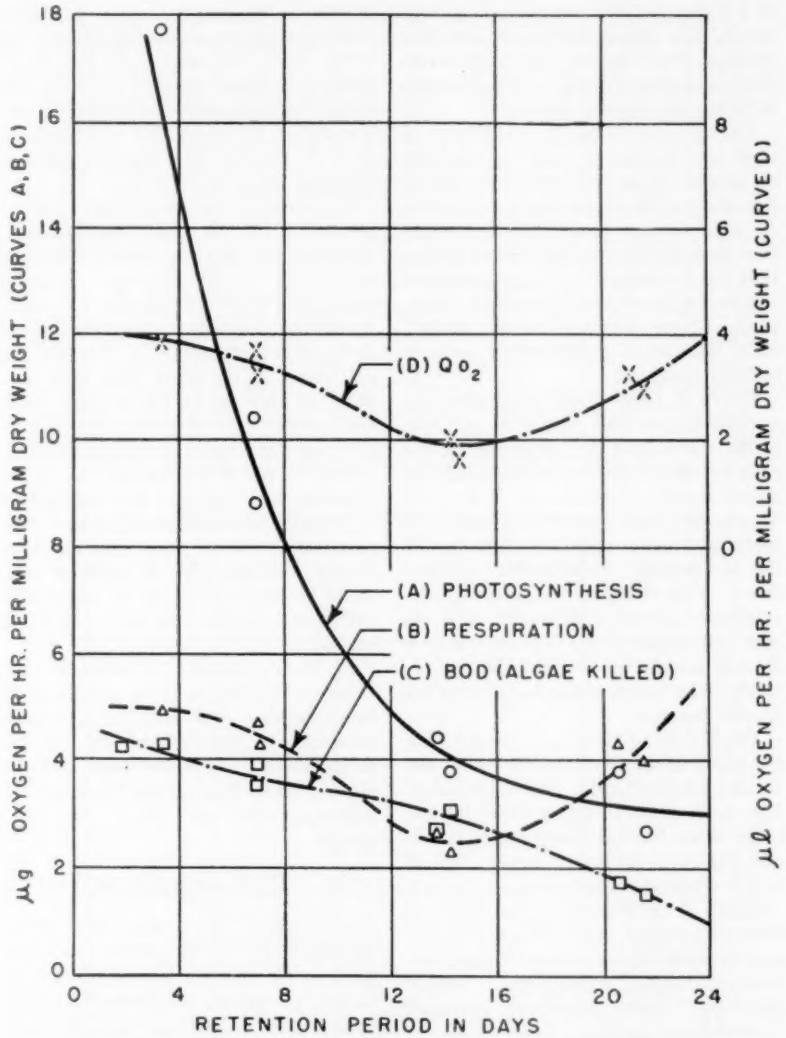

FIGURE 6.—Oxygen uptake and production capacities of *E. gracilis* at varying retention periods.

quirement (Curve G) of the algae in the absence of light—that is, Curve A less Curve D. Next the initial slopes of Curves C, F, and G were calculated, for each retention period studied. This was done by plotting the rates of oxygen exchange against elapsed time in the Warburg flask, and extrapolating to determine the initial rate. These data, which are assumed to represent the oxygen absorbing or releasing properties of the fresh culture sample,

at 250 foot-candles, are plotted in Figure 6. In more recent studies, employing recirculation instead of bubbling, dissolved oxygen within the tube is being measured directly.

Curves A, B, and C of Figure 6 show the uptake and also the release of oxygen in μg. per hour per mg. of dry algal cells in the culture. Curve A, representing total oxygen production within the Warburg flask, shows that the "younger" cells, or cells corresponding to shorter retention times, produce more oxygen than the "older" cells, because of their greater rate of cellular synthesis.

Curve B, illustrating respiration requirement per unit weight of cell material, shows that this decreases as the cells become larger and accumulate inactive stored materials, up to $R = 14$. Thereafter the respiration requirement increases, probably because of the increasingly unfavorable environment. This changes the nature of the cellular material synthesized and its rate of respiration. That such a change occurs beyond $R = 14$ has already been indicated in the discussion of cell density.

The B.O.D. of the whole culture with the algae dead, shown in Curve C, decreases with cell age. This is probably due to a higher fat content in the older cells, the fat being more stable and therefore having a lower rate of B.O.D. than carbohydrates.

Curve D of Figure 6 is a replot of Curve B, except that it shows respiration in terms of microliters oxygen uptake rather than micrograms. This is the "Q_{O_2}" value commonly reported in biological studies.

The oxygen production and respiration data thus far presented refer to unit weights of algae and do not take into account the concentration of algae in the culture. Figure 7 presents such data; specifically, curves representing total oxygen produced by photosynthesis and total respiration. The ordinate or difference between the two curves is the excess oxygen produced during periods of photosynthetic activity, which is noted to be greater at lower retention periods, due to the higher concentrations of rapidly growing cells. The very old cells, beyond $R = 18$, require more oxygen for respiration than they produce.

In practice, the respiratory requirements must be met continuously, whereas the oxygen production will occur only in the daytime. If pond conditions were comparable to those producing mainly young cells ($R = 2$), and foot-candles = 250, but the cell count in the pond were only one-fifth as high as in the culture tube, the average rate of production of surplus oxygen would be approximately 1.0 p.p.m. per hour. Older cells would produce less oxygen, and beyond $R = 7$ would consume more oxygen than produced. These data indicate that young but not old *E. gracilis* cells could be responsible for the supersaturation of oxygen often found in pond effluents.

It is of interest to compare the amounts of oxygen being produced at foot-candles = 250 with the amounts produced in the culture tube, if the latter be figured on the basis that the algal growth follows the equation for photosynthesis of simple carbohydrates:

$$CO_2 + 2H_2O \rightarrow CH_2O + H_2O + O_2$$

Calculations show that the ratio, weight of oxygen produced (as measured in the Warburg flask) per unit weight of carbon synthesized (in the culture tube), does approach the theoretical value when $R < 7$. As the retention period increases beyond $R = 7$, the calculated ratio becomes progressively lower. This indicates that the young, rapidly reproducing cells contain much higher percentages of photosynthetizing protoplasm than the older cells growing under conditions where nutrients are limiting.

Biochemical Oxygen Demand

As previously noted, Figure 5 illustrates typical B.O.D. curves for a whole culture sample and for its algal and non-algal constituents. For simplicity, Figure 8 has been prepared to show B.O.D. values for only a single time period (the 120-hr. manometric B.O.D.) as related to varying retention periods.

The B.O.D. of the culture supernatant (that is, of residual soluble sewage material plus soluble organic compounds secreted by the algae) is low for all retention periods. Presumably the oxidation of sewage organic material by bacterial action is accomplished to a high degree, even at short retention periods. Through photosynthesis, part or all of the available CO_2 is converted to algal cell material, so that the over-all transformation is from sewage organic material to algae. Fortunately, the algae constitute in no way any hazard to the public health; and, as already noted, they are relatively stable and would exert a low oxygen demand upon receiving waters, even if dead. The cells might remain alive indefinitely when discharged to receiving waters. The new environment will determine whether the cells undergo any changes; that is, become younger, grow older, or die. Young cells might produce through photosynthesis more oxygen than consumed by respiration, but old cells would tend to settle out and, in

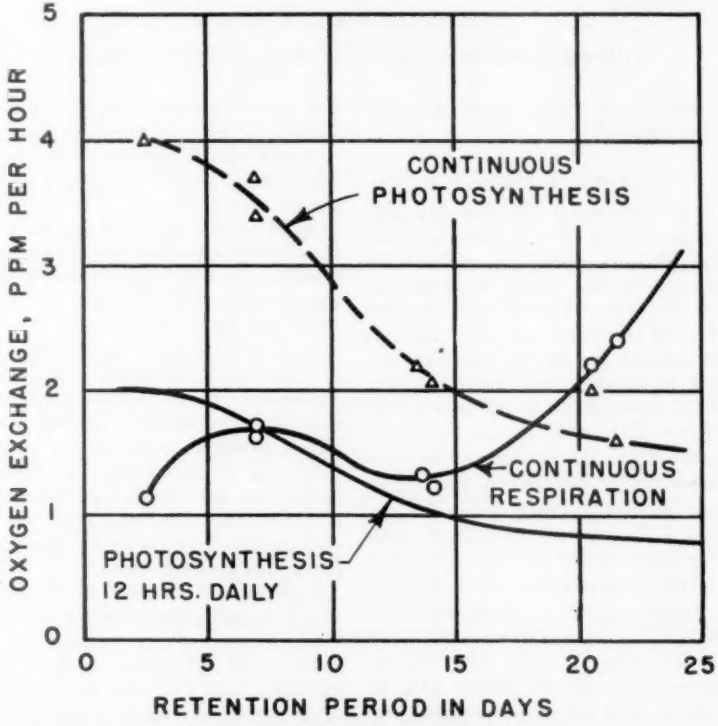

FIGURE 7.—Oxygen uptake and production capacities of cultures at varying retention periods.

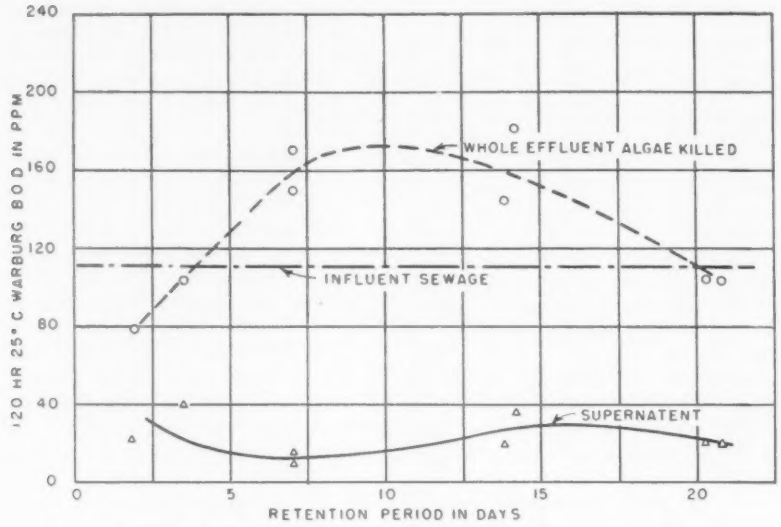

FIGURE 8.—Manometric B.O.D. of culture influent and effluent.

the absence of light, create a small oxygen demand.

Yield of Algae

It is interesting to compare the maximum yield of cell material produced when *E. gracilis* is cultured in sewage, with the yields reported by Cook (7) for *Chlorella pyrenoidosa* grown in synthetic media. In the latter studies the carbon was supplied in air enriched with 5 per cent CO_2, bubbled through the liquid; under this condition CO_2 was always in abundance. Also, fixed nitrogen, as ammonium ion or nitrate ion, and other minor nutrients were supplied as needed. The maximum yield, Y, was 0.48 g. dry weight per liter per day, at a cell concentration, D, of 0.36 g. dry weight per liter. (Note that $Y = D/R$.)

In the present Series III studies, ordinary air with no added CO_2 is employed, and the only available nitrogen and other nutrients are those initially present in the sewage. Under these conditions CO_2 is a limiting factor. A plot of yield of algae per liter per day (Figure 9) shows a maximum yield at the smallest retention period tested, $R = 2$. The maximum yield is 0.080 g. per liter per day, and the corresponding cell concentration is 0.160 g. per liter. This yield is small compared to Cook's value for *Chlorella*, due apparently to the shortage of CO_2 as well as to the slower reproduction rate of *Euglena* compared to *Chlorella*. With *Euglena*, the minimum feasible retention period, which allows sufficient reproduction time to build up a population, is approximately two days or slightly less. This is more than twice the retention period employed by Cook to obtain maximum yields of *Chlorella*.

Further information on this subject is available from the earlier Series I and II studies (1), wherein air enriched with CO_2 was employed for bubbling, and CO_2 was therefore less limiting. These data, together with the present information and those of Cook, are compared in Figure 10.

Apparently, the maximum yield of *Euglena* possible, with extra CO_2 added to permit maximum utilization of nitrogen, is 0.26 g. per liter per day. But by employing more rapidly reproducing algae, such as *Chlorella* or *Scenedesmus*, greater yields could be realized. Some recent experiments made with *Chlorella* and *Scenedesmus* show that these organisms are also at home and grow vigorously in a sewage environment.

This discussion indicates the economic possibilities of converting sewage organic materials to food. As compared with Cook's use of commercial CO_2 and nutrients, the use of sewage offers important advantages: (1) The expensive nutrient, fixed nitrogen, is available in the sewage, in the form of

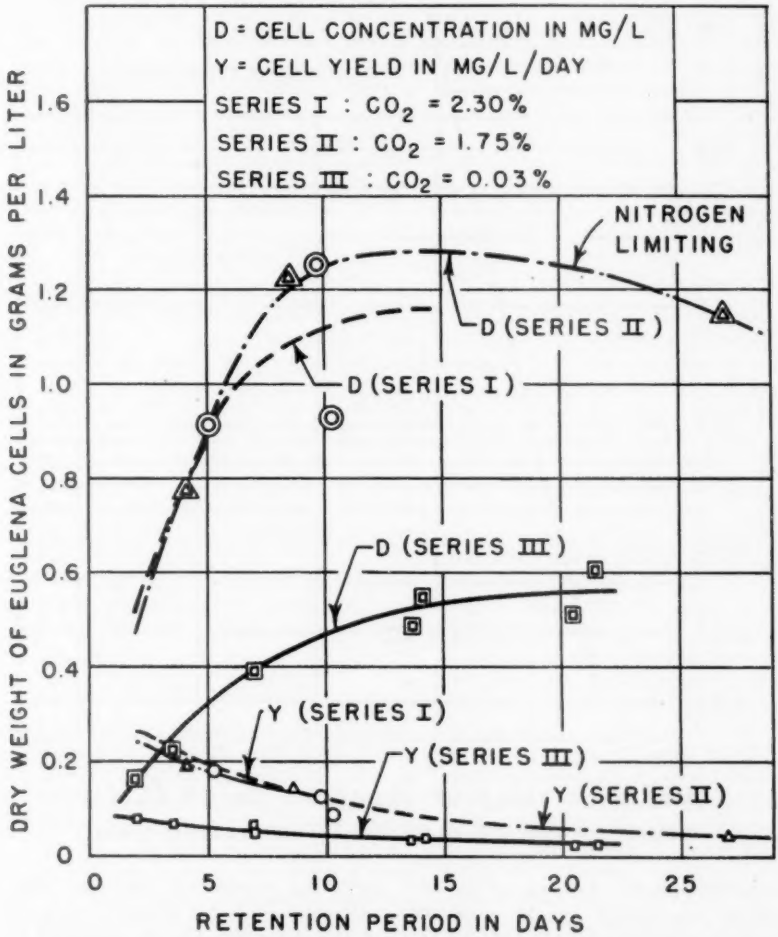

FIGURE 9.—Concentration and yield of *E. gracilis* at varying retention periods.

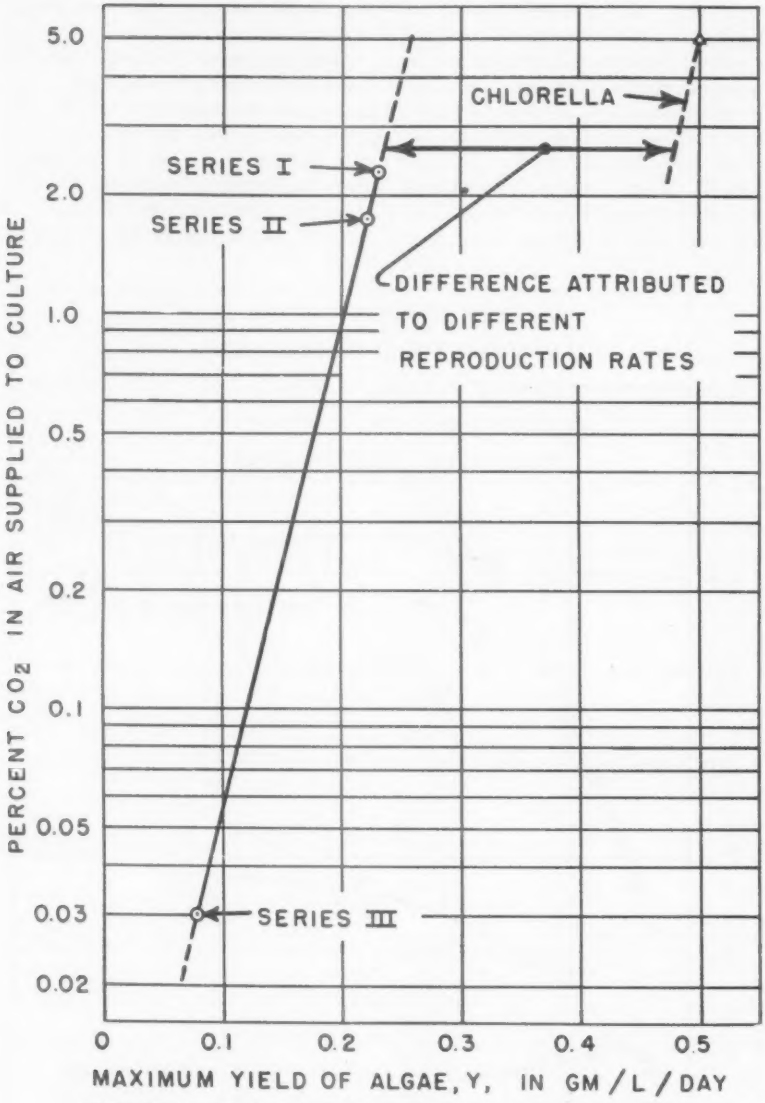

FIGURE 10.—Yield of *E. gracilis* related to available carbon dioxide.

ammonia initially present, or as ammonia or nitrate made available through continuing bacterial action; (2) other important minor nutrients, including phosphorus, are also originally present in the sewage; (3) much of the necessary CO_2 is produced in the sewage through bacterial oxidation

processes, which moreover may be maintained by the oxygen produced through algae photosynthesis. Also, sewage appears to contain no deleterious elements in any significant amounts; in fact, *E. gracilis* was found actually to prefer sewage to various artificial media specially prepared for this species on the basis of data reported in the literature (1). There appears no reason why the valuable organic nitrogen contained in settled sewage cannot commercially be reclaimed in the form of high-protein algae, and this employed as food, say for cattle, following its sterilization. (Separation of the algae from the liquid medium is readily accomplished by flocculation with alum, followed by settling.) "Sewage to beefsteak" may be a real economic possibility.

Alkalinity and pH

The titratable alkalinity of the settled sewage introduced into the culture tube each day was approximately constant at 50 p.p.m., with pH of 6.5. At $R = 2$ the alkalinity drops to 10 p.p.m. and the pH to 5.3. At $R = 3$ the alkalinity decreases to zero and the pH to 4.3. In the range $R \geqq 5$, the pH remains approximately constant at 3.2. Such low pH's, as shown by Jahn (8), do not necessarily inhibit the growth of *Euglena*.

Because of the shortage of CO_2, the algae abstract carbon from the alkalinity of the sewage, in accordance with the reaction

$$HCO_3^- \rightarrow CO_2 + OH^-$$

This reaction alone does not effect any change in alkalinity, and furthermore would cause an increase in pH. It must be concluded that the algae produce some strong acid, or else release H^+ ions (perhaps in exchange for absorbed NH_4^+ ions), in amounts more than enough to neutralize the hydroxyl ions produced, thus destroying the alkalinity and lowering the pH. (The destruction cannot be attributed to precipitation of $CaCO_3$, since precipitation can occur only as a result of an increase in pH.) Measurements were made of the acidity (by titrating with NaOH to pH 4.3) of the algae-free culture supernatant. Examination of these data shows that in the range $R = 3$ to $R = 20$, the acidity increases in proportion to retention time, at an increment of 2 p.p.m. per day.

Some recent studies (to be published later), made under the same conditions except that the sewage alkalinity was higher (115 p.p.m.), also show a reduction in alkalinity but an increase in pH. In this case a considerable alkalinity remains, indicating that the amount of acid produced is less than the amount of hydroxyl ion released, so that although some of the alkalinity is destroyed the pH nevertheless increases.

Materials Balance

It is important to compare the amounts of cell organic material synthesized with the amounts of organic material introduced into the culture tube. Figure 11 shows a plot of the carbon contained in the algae cells, compared with the amounts of carbon made available to the culture. (The carbon and hydrogen contents of the algal cells remained approximately constant at all retention periods. The average values, in per cent of dry cell weight, were 45.9 for C and 6.46 for H.) For $R \geqq 3.5$, more carbon is contained in the cells than is contained in the introduced sewage, and the difference must be abstracted from the bubbling air. Although the solubility of free CO_2 in water at equilibrium with ordinary air is very low (about 0.5 p.p.m.), the continuous utilization of free CO_2 by the algae permits continued solution of CO_2 from the bubbling air. The rate of pick-up of CO_2, indicated by the slope of the curve, is rapid and approximately constant for the younger, rapidly growing cells, and decreases as the cells become older

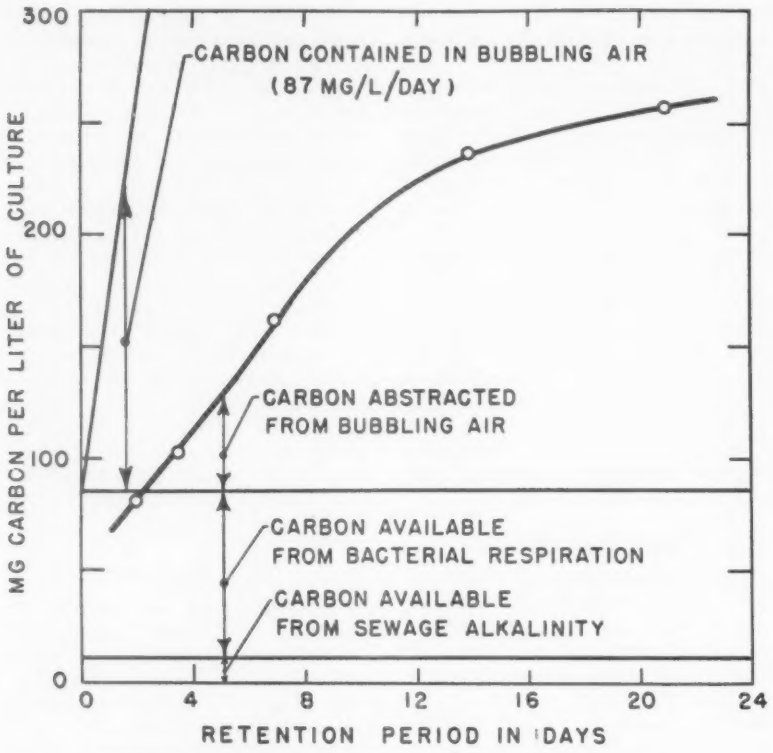

FIGURE 11.—Carbon balance for *E. gracilis* cultured in sewage.

and grow more slowly. The total amount of CO_2 abstracted is greater for the older cells, because of their greater total mass.

The amount of CO_2 available to the algae from the bubbling air increases with retention time. Hence, in the Series III tests, it appears that free CO_2 is the principal factor limiting cell population for $R > 7$, up to some long retention period beyond which CO_2 becomes more available and the mineral nutrients become more limiting. In the Series I and II tests, the culture pH was 7.2 and alkalinity 95 p.p.m., and the equilibrium free CO_2 concentration was of the magnitude of 500 to 1,000 p.p.m. In these tests free CO_2 was not limiting, and the brownish discoloration of the cells at long retention periods may have been due to over-exposure to CO_2 or "burning."

The data presented in Figure 12 refer to the algae cells, and do not account for any synthesized materials which may be secreted by the cells into the liquid medium of the culture. There are several evidences of such materials, including: (a) A small amount of mucilage accumulates within the culture tube, mostly adhering to the tube wall above the water line. The amount formed increases with increasing retention period. In the Series I and II tests (1), the amounts formed were very great; hence, the amount formed increases with the availability of CO_2. (b) For $R \geqq 7$,

the dry solids obtained by evaporating the algae-free culture contain an aromatic compound, characterized by a sweetish cocoa-like odor. From $R = 7$ to $R = 20$ the intensity of odor increases with retention time, from faint to very strong.

Inorganic Ion Absorption

Figure 12 comprises sets of curves which illustrate the absorption of certain inorganic ions during photosynthesis; specifically, Ca, Mg, K, Mn, NH_4, PO_4, and also total ash. Two curves are shown for each of these constituents, one representing the amount of constituent contained in a unit of dry algal cell material, and the other the amount of the constituent made available in the culture per unit of dry algal cell material. In both cases the amounts are expressed as a percentage of the dry algal cell weight.

Nitrogen

The nitrogen content of the *Euglena* remains approximately constant, between 5 and 6 per cent. The ammonia available to the *Euglena* is sufficient to provide for its nitrogen requirements up to about $R = 14$. Beyond $R = 14$ the culture contains considerable nitrate nitrogen, apparently resulting from bacterial action; but it is not known whether the *Euglena* can utilize this type of nitrogen. Hence nitrogen may be a limiting factor beyond $R = 14$.

Phosphorus

The phosphorus requirement is greatest for the youngest and most rapidly growing cells, indicating that phosphorus utilization is closely associated with photosynthesis. The curves indicate that phosphorus is not limiting; but, if it is considered that not all of the measured phosphorus is actually available to the organism, then it is possible that phosphorus may be limiting when $R > 7$, where the two curves become parallel.

The concentration of phosphorus in the added sewage, 25 p.p.m. as PO_4, is perhaps slightly higher than normal values for sanitary sewage.

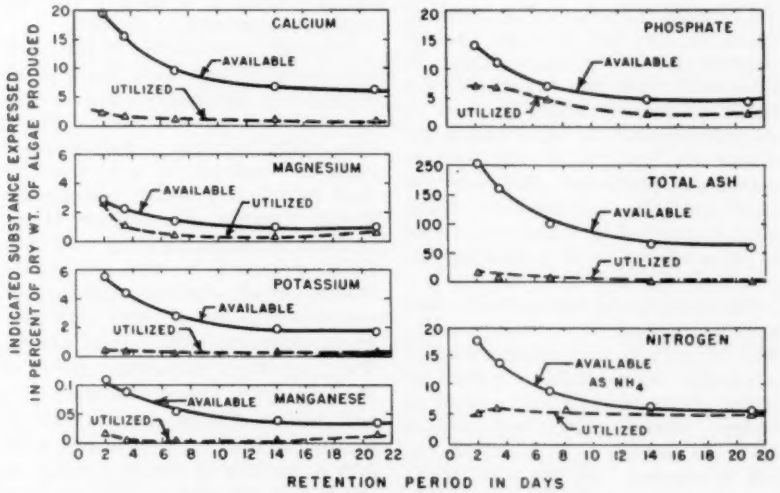

FIGURE 12.—Absorption of ions by *E. gracilis*.

Magnesium

These two curves are of interest because they converge at $R = 2$. Hence, the availability of magnesium at very low retention periods may be a factor limiting reproduction in that range. Were greater amounts of magnesium made available, the maximum yield of algae may have been greater.

Conclusions

In regions where the climate is sunny and dry, oxidation ponds afford a simple and economical means for biological stabilization of settled sewage and industrial wastes. Their advantages of low construction cost and, more important, very low labor requirement for operation, are becoming increasingly apparent to sanitary engineers.

The treatment effected by an oxidation pond results from a complex symbiosis of bacteria and algae. The principal work of treatment is accomplished by aerobic bacteria, which oxidize the organic carbon contained in the sewage to carbon dioxide. The algae, through photosynthesis, convert much of this carbon dioxide to algal cell material. Therefore, the pond effluent may contain as much organic material as the sewage entering it. Whereas the entering sewage solids are highly putrescible and hazardous to the public health, the algae cells in the effluent are highly stable and have no pathogenic significance. Moreover, the algae constitute a living food, which when discharged to watercourses may be of much value in promoting the growth of fish and other wildlife.

The properties of an algal cell—that is, its form; shape; size; density; color; rate of growth and photosynthesis; respiration rate; vitality; content of carbohydrate, fat, and ash; content of various mineral ions; etc.—depend upon the environment to which the cell is exposed. In the case of *E. gracilis*, cells exposed to favorable conditions (an abundance of nutrients and light) are small, dark green in color, highly motile, rapidly reproducing and growing, contain a minimum of fat, and photosynthesize much more oxygen than they respire. These may be designated "young" cells. "Old" cells are those growing in an unfavorable environment. They are mostly in a passive (palmellar) state, have little motility, are large and fat, are yellowish in color due to lack of chlorophyll, are reproducing and growing very slowly, produce less oxygen than they respire, and tend to settle out and adhere to bottom surfaces.

The types of cells contained in pond effluents are principally young cells, producing much more oxygen than they use up in respiration. When discharged to watercourses they may remain alive almost indefinitely. The new environment will determine whether the cells remain young or become old, but it appears that they cannot become old until the stream waters have become clean and practically free of pollution. Thus, in polluted streams, where oxygen is most critically needed, the algae will be producing a surplus of oxygen; only in clean streams where oxygen is abundant, will the algae create an oxygen demand.

The algae growing in the ponds absorb ammonia and other forms of nitrogen from the sewage and thus may be considered a means for reclaiming this waste nitrogen. Actually, the algal cell material is a high-protein food, which is probably suitable for feeding to cattle. The commercial possibilities of producing algae food from sewage appear to be good, because the sewage already contains nutrients necessary for growing algae, and contains nothing to inhibit their growth. The probable maximum yield of *Euglena* from sewage, fortified only with carbon dioxide and subjected to continuous lighting, is estimated to be 0.26 g. per liter of sewage per day, or about half the yield which has been obtained with *Chlorella* cultured in

ideal synthetic media prepared from commercial nutrients. Fortification with carbon dioxide is necessary to permit maximum reclamation of the available nitrogen. By using a rapidly reproducing alga, such as *Chlorella*, the yields obtained from sewage can probably be increased to some higher limit.

New series of investigations are being made with the laboratory culture tubes, to study further the behavior of *Euglena* and also of other species of algae, with sewage bacteria both present and absent. Pilot-scale outdoor studies are also planned, to make possible a direct comparison of laboratory and field data. The pilot studies will be made with an outdoor trough operated to simulate conditions occurring in actual ponds.

Acknowledgments

Appreciation is extended to Professor W. F. Langelier, Professor Harold Kirby, and Mr. A. Greenberg of the University of California; Senior Sanitary Engineer Paul Ward of the State Health Department; Mr. David H. Caldwell, consulting engineer; and Mr. Keeno Fraschina, of the City of San Francisco. Their cooperation has greatly facilitated this work.

This investigation was supported by a research grant from the National Institutes of Health, U. S. Public Health Service.

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EFFECT OF PARTICLE SHAPE ON POROSITY AND SURFACE AREA OF TRICKLING FILTER MEDIA

By George J. Schroepfer

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Historical

Trickling filters have been employed in the treatment of sewage for approximately one-half a century. In the United States this method was incorporated in 1,486 plants, or approximately 50 per cent of the communities providing secondary treatment as of 1942.

In this process sewage, after being subjected to preliminary treatment, is sprayed upon beds of filter media, in which under a favorable environment biological processes partially stabilize the organic matter contained therein. The media commonly employed have been crushed stone, gravel, or slag. Because of the requirements or economics of the situation, crushed granite, quartzite, trap rock, or gravel are usually provided in the Midwest.

As would be expected because of the widespread use of this method of treatment, and the fact that a large proportion of the cost is represented by the media employed, much attention has been given to durability, size, and cleanliness of the material to be provided. Also, the subject of this investigation—that is, the effect of flat and elongated particles—has not been neglected.

Unfortunately, the writings on the subject have been chiefly in the form of general statements or of specifications ruling out summarily, or by specific requirements, material containing an excess of such shapes. Nothing in the form of a basic investigation has come to light. Specifications range from those which completely ignore the subject to those which provide in considerable detail the percentages of various shape ratio material which will be tolerated. A review of coarse aggregate specifications for concrete would indicate that many of the filter media requirements as to particle shape may have had their origin in specifications for this type of material.

Historically, the earliest reference found on the subject dates to 1908 in the requirements for media on the largest trickling filter installation ever to be constructed in the United States, at Baltimore, Md. This specification (1) provided:

"The filtering material shall be composed of a stone which breaks or crushes in approximate cubes, or in pieces which are approximately equidimensional. The presence of more than 10 per cent of flag pieces or slabs will be deemed sufficient cause for rejection of the entire lot of broken stone. Any piece of stone will be considered a slab which has two approximately parallel faces, the average of whose areas is more than five (5) times the distance between the approximately parallel faces."

Similar requirements persist in later specifications. The Federal Specification (SS-C-744a-Dec. 15, 1949) for sewage trickling filter provides:

"The individual pieces of the filter media shall be as nearly equi-dimensional as practicable. Elongated pieces, flat pieces, or both, occurring in the media shall not exceed 10 per cent of the total by weight, and materials having a marked tendency to crush to such shapes shall not be used. For

purposes of this requirement, elongated pieces and flat pieces are defined, respectively, as having length exceeding the width and width exceeding thickness in ratio of more than 1.5 to 1.0, the dimensions being the maximum in the piece under consideration."

A still later publication (2) contains the following statement: "The media should be approximately cubical in shape and free from flat or elongated pieces."

Numerous other specifications have been examined, most of which contain a general reference—oftentimes ambiguously or indefinitely worded—to flat or elongated pieces. Difficulties have resulted because, on the one hand, industry has not known in bidding what material would be required by the supervising engineer; on the other hand, the engineer has not known how rigid he should be in view of the uncertainties of the situation. Indefiniteness on underlying principles has led to confusion.

As a result of the concern of one consulting organization in particular, and of several meetings with other consulting firms and representatives of producing industry, the Dresser Trap Rock Company of Minneapolis, Minn. sponsored a research project to be undertaken by the Sanitary Engineering Division, Department of Civil Engineering, University of Minnesota, designed to collect certain basic information on the effect of this factor.

Although the investigation was necessarily limited in scope, it was designed to include basic information on two factors, of apparent consequence. One was on the effect of shape factor on the percentage of voids in the media. The other was on the effect of this factor on surface area. It is generally conceded in practice that a high percentage of voids, and a large surface area per unit of volume are conducive to efficient filter operation.

Purpose and Scope

As previously stated, this investigation was undertaken to determine the effect of flat or elongated particles on the voids and surface area of the material. In general, for the same shape material the voids do not vary appreciably with size for a uniform grading. Therefore, any variation in voids will be caused by changes in the shape of the material. Considering the surface area, it would be expected that this factor for a material of the same size and grading should increase to some extent as the shape ratio increases.

Primary considerations of this paper are, therefore:

1. The relationship between surface area and shape ratio.
2. The effect of shape ratio on the unit weight and, therefore, on the voids.
3. The effect of the median particle size—for a given shape ratio—upon the surface area and voids.

The material studied in this investigation is limited to trap rock from the Dresser, Wis., quarry of the Dresser Trap Rock Company of Minneapolis, Minn. The quarry location is east of the St. Croix River, approximately three miles south of St. Croix Falls, Wis. and one mile north of Dresser, Wis.

Definitions

Shape ratio—A measure of the shape of a stone determined by dividing the greatest major dimension (length) by the least dimension perpendicular to the long axis (thickness) in expressing the degree of flatness or elongation.

Cubical shape—A perfectly cubical piece would have a shape ratio of 1.0; in this report shape ratios ranging from 1.0 to 2.0 were considered as cubical or low shape ratio material.

Voids—The percentage of the volume not occupied by solids; total volume minus volume of solids equals

volume of voids; therefore, per cent voids equals volume of voids divided by total volume.

Surface area—The surface area of the rock in a unit volume of material. In this report the unit surface area in square feet per cubic foot is employed.

Apparatus

A box 18 in. by 18 in. by 18 in. (3.375 cu. ft.), made of ⅝-in. plywood was used as the known volume in determining the unit weights of each rock sample. The use of a greater than 1 cu. ft. volume was selected to reduce the errors obtained by void pockets and surface effects of the container. All unit weights were determined and recorded to the nearest 0.5 lb.

After considering a variety of coatants to determine surface area, a flat paint, selected after consultation with paint chemists, was used to coat the samples by immersion. The paint was quite viscous (at room temperature) so that a thick coating was retained on the rocks. This caused an appreciable weight of paint to be left on the rock (about 0.4 lb. per square foot of surface).

A wire screen (1-in. square holes) was used to dip the rocks into the paint. This permitted the excess to drip off during a controlled time interval. The paint was held in galvanized buckets approximately 12 in. in diameter and 8 in. deep.

Methods and Procedure

Except for one sample collected from the South St. Paul trickling filter, the rock used was taken from stock piles at the Dresser, Wis., quarry. Ten bags (about 0.5 ton) of various shape ratio material were selected by hand, in addition to 18 bags (about 1 ton) of unselected plant-run material obtained by quartering. Four control rocks of somewhat regular surfaces and an area which averaged about 1 sq. ft. each were selected. The regular surfaces facilitated the measurement of the surface area of these rocks.

Except for two plant-run samples, all the material was graded according to shape ratio, which was obtained by the measurement of the long and short axis of each individual rock. The short axis was taken as the least dimension perpendicular to the long axis. The long axis was considered to be the largest dimension of the rock. Shape ratio ranges were arbitrarily taken as 1–2, 2–3, 3–4, and 4–5. These different shape ratio samples, two plant-run samples, and a sample from the South St. Paul sewage treatment plant, composed the different samples studied. It is probable that the median shape ratio for some of the shape ratio samples is not the average of the range; for example, shape ratio sample 1–2 will in all probability have a median shape nearer 2.0, because of the fact that there are very few perfectly cubical shapes.

Each shape ratio was then weighed in the box of known volume and the unit weight in pounds per cubic foot was obtained.* The two samples of plant-run material obtained by quartering were also weighed in the same manner. To reduce errors due to different placement of rock into the box, an average of at least three independent measurements (by three observers) of each sample was used as the unit weight of the sample. In all cases, the weight per unit volume is the weight obtained by loosely shoveling the rock into the box.

The plant-run material and the South St. Paul plant material was graded according to shape ratio and both the weights and number of each ratio were recorded.

All samples except the South St. Paul plant material were sized and the weights of each size were recorded. The sizing was done by hand-fitting the rocks into square holes of 2-in., 2½-in., 3-in., 3½-in., 4-in., 4½-in.,

* Standard method [ASTM Designation (29–42)].

and 5-in. openings cut in a piece of ¼-in. plywood.

For control purposes, four large rocks of about 15 lb. each whose surfaces were quite flat were measured for surface area. The surfaces were divided into rectangles and triangles and the areas of these polygons were recorded. Also, seven rocks selected from the plant-run material because of flat and regular surfaces were measured for surface area. This was done by planimetering the trace of each of the surfaces of the rocks. All areas were recorded in square feet. Measurements were made independently by three different investigators and the results averaged.

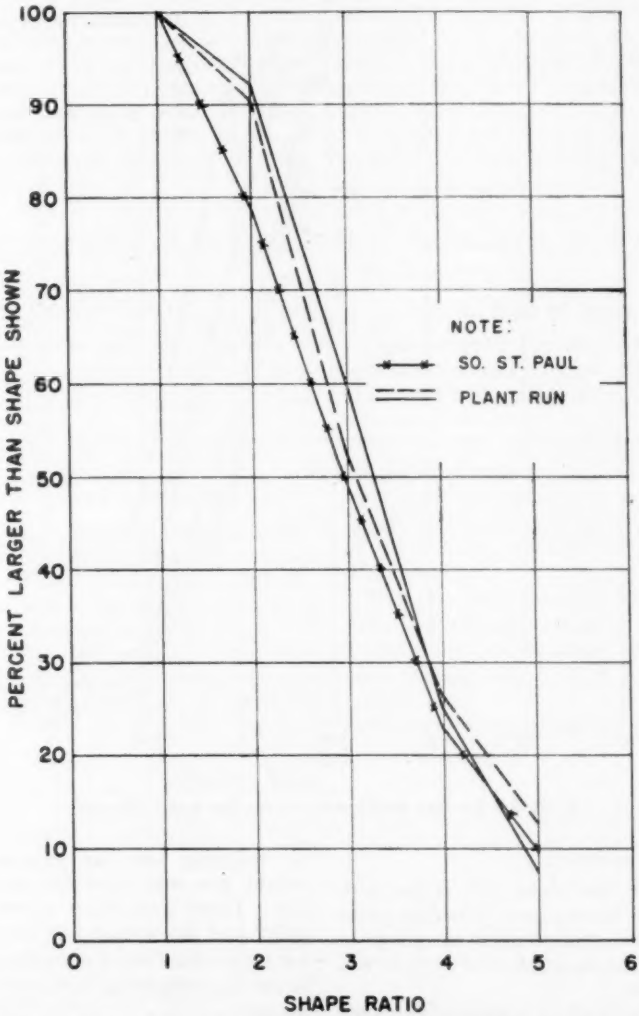

FIGURE 1.—Shape distribution curves for plant-run and South St. Paul plant samples.

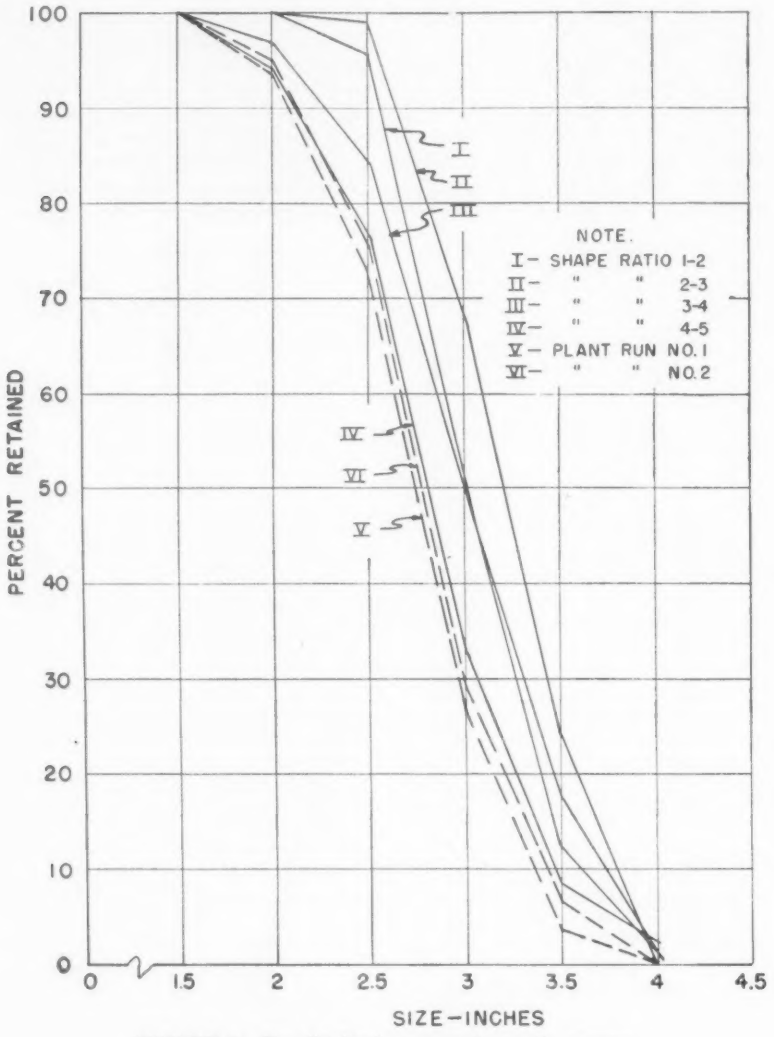

FIGURE 2.—Size distribution curves for media samples.

Paint Coating

The control rocks were coated with paint by the standard procedure given below and the weight of paint in pounds per square foot of surface was obtained.

A cubic foot of material from each of the various samples was obtained by weighing out the average unit weight per cubic foot for each sample. These were then coated with paint and the square feet of surface for each cubic foot of material was obtained by comparison with the control samples.

The following procedure was stand-

ardized and used in coating. The bucket in which the rock was to be dipped was two-thirds filled with paint and weighed, along with the 1-in. wire screen, to the nearest 0.01 lb. (The inclusion of the screen weight prevented any error from the weight of paint adhering to the screen.) Five to nine rocks at a time were placed on the screen, completely submerged in the paint, and then allowed to drip into the paint bucket for one minute. The process was repeated until 1 cu. ft. of that sample had been run. The bucket of paint, plus screen, was then weighed and the loss of weight of paint determined by subtracting the final weight from the initial weight.

The 1-min. dripping period allowed most of the excess paint to drain off and yet did not allow the paint to dry. Care was taken in placing the rocks on the screen so that paint was not caught between the surfaces of two rocks.

A rerun of samples of shape ratio of 1–2 and 3–4 indicated the tests could be duplicated with an error of 2 per cent or less.

Data for the Shape Relation

Figure 1 presents the shape distribution of the two plant-run samples and the South St. Paul plant sample. The median values of the shape ratio were determined from the weights of material in the various shape ratio groups. The median values for the two samples of plant-run material are 3.29 and 3.09, and the sample of material from the South St. Paul sewage treatment plant has a median shape ratio of 2.98. For the plant-run material the extremes vary from a ratio of 1 to more than 5. The interquartile range (middle 50 per cent) varies from 2.5 to 4.0.

The samples of material examined have a high shape ratio compared to the usual thinking of cubical shapes as expressed in specifications. Although the large-sized plant-run material has a slightly higher shape ratio than the smaller-sized material (maximum size 2 in.) taken from the plant in South St. Paul, it is surprising that the plant-run material apparently has a shape ratio distribution quite similar to the much smaller South St. Paul plant material.

Data on Size of Material

Figure 2 gives the size distribution for the various shape ratios. The South St. Paul plant sample was not sized, but was considerably smaller than the other material. (Several of the largest pieces of rock from the South St. Paul plant were sized and it was estimated that approximately 5 per cent of the material would be retained on a 2-in. screen.)

The plant-run material had a median size of approximately 2.76 in., whereas the various shape ratio samples ranged from 2.82 to 3.2 in. in median size.

TABLE I.—Characteristics of Various Samples

Sample	Av. Unit Wt. (lb./cu.ft.)	Median Size (in.)	Average Voids (%)	Surface Area (sq.ft./cu.ft.)	
				Run 1	Run 2
Shape Ratio 1–2	95.6	3.02	49.0	15.8	15.8
Shape Ratio 2–3	93.4	3.20	50.3	14.7	—
Shape Ratio 3–4	89.8	3.0	52.2	14.7	15.0
Shape Ratio 4–5	85.0	2.82	54.7	19.2	—
Plant run No. 1	87.1	2.77	53.6	17.0	—
Plant run No. 2	85.3	2.75	54.5	17.3	—
South St. Paul	93.0	—	50.4	—	—

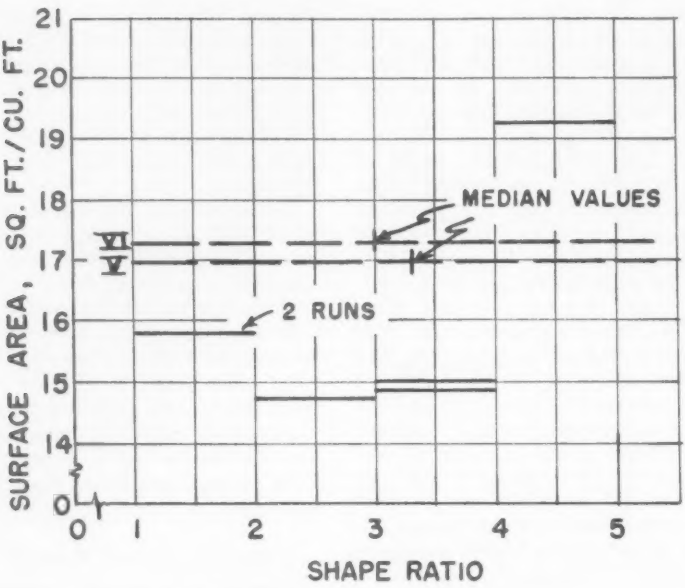

FIGURE 3.—Relationship between area and shape ratio.

Data from Coating Procedure

Two of the large rocks were coated separately by the standard coating procedure. The coating obtained for each was 0.370 and 0.407 lb. of paint per square foot, respectively. Of the smaller stones, four were coated by the standard procedure and a coating of 0.348 lb. of paint per square foot was obtained.

The four smaller pieces of material were thought to be more representative of the samples of material to be coated (having similar size, fractures, and roughness); therefore, they were used in determining the surface area per cubic foot of the four shape ratio samples and the two plant-run samples. Because the comparison of the surface areas of the various shape ratios is a relative matter, the control tests were not duplicated as a check.

Table I gives the results obtained in square feet of surface area per cubic foot of material. Figure 3 shows the various shape ratios and the two plant runs (lines V and VI) plotted against surface area. The crosses on the lines representing the two plant runs approximate the median shape of the material.

The data on unit weights of the various materials are summarized as follows:

	Shape Ratio				Plant Run		South St. Paul Plant
	1-2	2-3	3-4	4-5	No. 1	No. 2	
No. of measurements	4	5	10	4	4	5	3
Av. unit weight (lb.)	95.6	93.4	89.8	85.0	87.1	87.3	93.0
Voids (%)	49.0	50.3	52.2	54.7	53.6	54.5	50.4

The relationship between shape ratio and unit weight is plotted in Figure 4.

Summary and Conclusions

Although the investigation reported herein was not sufficiently inclusive to permit of the making of broad generalizations and definite conclusions, it was performed—for one type of material—with sufficient care as to details, as to make possible the following summary statements:

1. The relationship between shape ratio and surface area is not readily apparent from the information already presented. Figure 3 illustrates this point. However, the relationship in Figure 3 is masked by another factor, the size effect. Figure 2 shows that the median sizes of the various samples varied from 2.75 to 3.2 in. Because the surface area varies inversely with size, this factor must be evaluated. If spherical particles are considered, S, the surface area in square feet per cubic foot of media, is equal to 39.6 divided by D, the diameter in inches of the media, for an assumed 45 per cent voids. Using this relationship, the observed surface areas can be corrected in an approximate way for the size factor by assuming uniform size spherical particles of the median diameter of the irregular shaped particles in the samples. In the following calculations, the surface areas are corrected to the average median size of the plant-run material (2.76 in.):

It will be seen that the corrected surface areas do not vary appreciably with shape ratio. The average of the surface areas for a shape ratio range from 1 to 5 is 17.8 sq. ft. per cu. ft., as compared to an average of 17.5 for the plant-run material. Theoretically, the surface area should increase slightly with an increase in shape ratio. Thus, a cube 3 in. on a side has a surface area of 54 sq. in. whereas the same volume in a rectangular parallelepiped 6 in. by 3 in. by 1.5 in. (shape ratio of 4) has a surface area of 63 sq. in. This involves an increase of about 4 per cent for an increase of 1 in the shape ratio range.

Investigation by others (3) has shown that the B.O.D. reduction by a filter increases slightly with an increase in surface area of the media. As an illustration, this work indicated that a major increase in the surface area (from 20 to 80 sq. ft. per cu. ft.) increased the per cent reduction in B.O.D. by about 8 per cent, or about 1⅓ per cent for an increase of 10 sq. ft. per cu. ft. in the surface area. The greatest range in observed surface area for all the shape ratios considered herein was from 14.7 to 19.3 sq. ft. per cu. ft., indicating a maximum possible effect of this factor of less than 1 per cent. The above would indicate that from a theoretical standpoint the shape factor has very little effect on the surface area and, hence, the efficiency of a trickling filter. However, from a practical standpoint, as will be discussed later, there is a tendency

Sample	Shape Ratio Range	Median Size (in.)	Surface Area (sq.ft./cu.ft.)		
			Observed	Based on Spheres	Corrected[1]
I	1–2	3.02	15.8	13.15	17.3
II	2–3	3.20	14.7	12.40	17.1
III	3–4	3.0	14.9	13.20	16.3
IV	4–5	2.82	19.3	14.10	19.7
Av.	—	—			17.8
V	3.29	2.77	17.0	14.4	17.5
VI	3.09	2.75	17.3		

[1] Based on median sphere size of 2.76 in. as a standard.

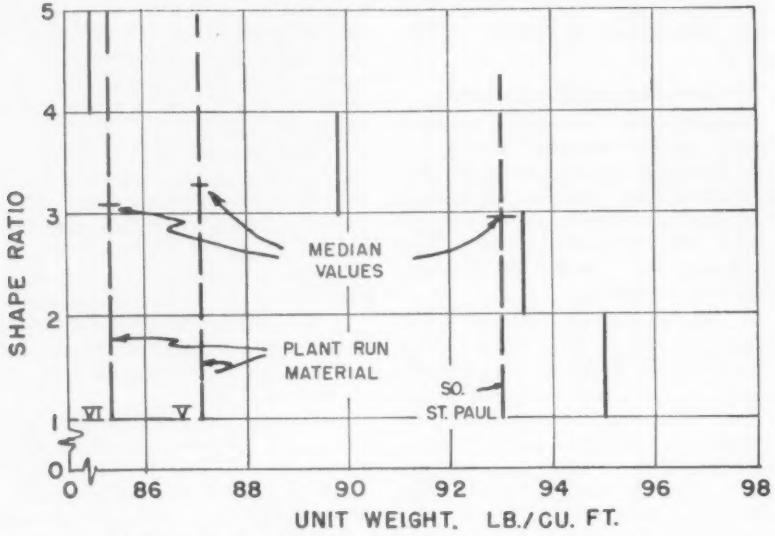

FIGURE 4.—Relationship between shape ratio and unit weight.

for pieces of high shape ratio material to "pile" on one another on the flat sides; therefore, the entire surface area measured above may not be effective. This "piling" may have the effect of increasing the effective size of the media.

2. The investigation reveals a definite reduction in the unit weight—and, therefore, an increase in the percentage of voids—with an increase in shape ratio. For summary purposes, this is illustrated in the following table:

Shape Ratio Range	Av. Unit Wt. (lb./cu.ft.)	Average Voids (%)
1–2	95.6	49.0
2–3	93.4	50.3
3–4	89.8	52.2
4–5	85.0	54.7

It is desirable in trickling filter design and operation that as much void space as possible be provided to facilitate ventilation, unloading, etc. The results of this limited investigation would indicate that an increase in the shape ratio would be advantageous from this aspect.

It was considered desirable to relate the above data to an actual plant employing the same type of media. For this purpose the South St. Paul plant, which has been operating without ponding difficulties for more than ten years at very high loadings, was selected. Although of smaller size than the material tested herein, analysis showed that its median shape ratio was also relatively high (2.98) and that its unit weight of 93.0 lb. per cu. ft. agreed quite closely with the data obtained herein, as a comparison with the above summary table will indicate.

3. To offer some visual indication of the appearance of the media in place, a plexiglass front was installed on the unit weight box, and photographs taken, for the extreme shape ratio ranges (1–2 and 4–5), as well as for the plant-run material. The material was placed in random style by shovel-

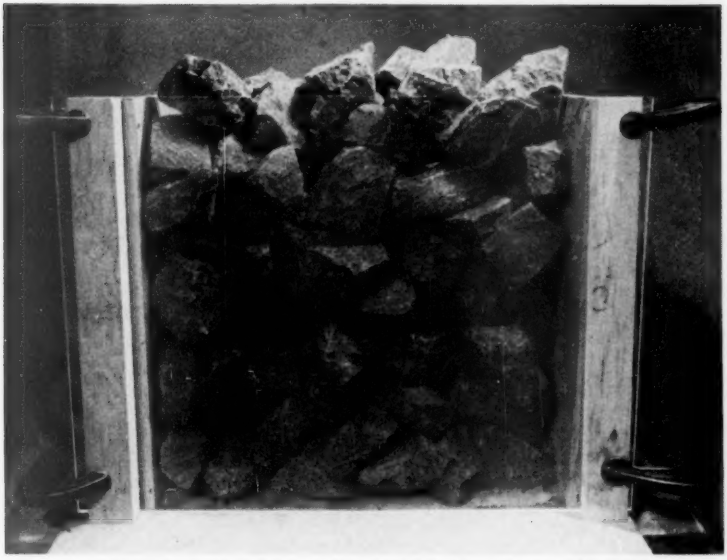

FIGURE 5.—Appearance of material having shape ratio 1–2.

ing into the box. Shape ratio 1–2 (Figure 5) appears to have uniform void spaces, as would be expected. Plant-run material (Figure 6) shows a uniform dispersion of high and low shape ratio material, with the voids being relatively uniform in size and no apparent "piling." Shape ratio 4–5

FIGURE 6.—Appearance of plant-run material.

FIGURE 7.—Appearance of material having shape ratio 4–5, indicating tendency to "pile" on flat surfaces.

(Figure 7) indicates a definite tendency of "piling," with the formation of large void pockets. Figure 7 would support the concern of many specification writers over too great a percentage of flat and elongated pieces. However, it must be remembered that this material was composed of practically all "flats" of very high shape ratio. Even for the plant-run material of high median shape ratio (3.09 and 3.29) the tendency to "pile" was not marked. That this tendency does not occur to a degree to affect operation of the filter is shown by the data on the high ratio material at the South St. Paul plant supported by plant operating results.

4. It would be desirable to supplement this work at a later date by collecting similar data on other filter media, such as granite, quartzite, and gravel. To permit of this, the procedures followed have been outlined in considerable detail herein.

Acknowledgments

This paper is a condensation of a report on an investigation conducted at the Sanitary Engineering Laboratory, in the Oak Street Laboratories of the University of Minnesota, under the supervision of the author. The work was directed by Lewis P. Carpenter, graduate student, who was assisted by Richard G. Kilp and William B. Hamre, senior civil engineering students at the University of Minnesota.

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CHEMICAL TREATMENT OF SEWAGE FOR SHORT PERIODS *

By Ward E. Conrad

Chief Operator, Sewage Treatment Plant, Wooster, Ohio

In 1939, Wooster, Ohio, placed in operation a sewage disposal plant incorporating the use of chemical precipitation during periods of the year when the receiving stream could not satisfactorily assimilate the effluent from plain sedimentation operation.

For background the following facts are of interest. The plant was designed for a population of 19,300, as of 1960, with a maximum sewage flow of 6 m.g.d. and an estimated 20-day B.O.D. loading of 0.25 lb. per capita per day. The present population of approximately 14,000 and an average flow of 2.283 m.g.d. with a 3.950-m.g.d. maximum (1950) gives an operation well under capacity design. In the 11 years and 7 months of operation to the end of 1950 the suspended solids reduction reached a high of 73.9 per cent (7 months in 1939) and a low of 60.1 per cent (1943). The B.O.D. reduction has been as high as 44.3 per cent (1939) and as low as 29.9 per cent (1943). The low in 1943 was 8.6 percentage points less than the next lowest year.

Layout and Units

A flow diagram of the plant (Figure 1) shows the raw sewage influent going from a control chamber equipped with a shutoff gate, to a 25-in. comminutor and a stand-by emergency bar screen, and thence to the wet well, where the sewage is lifted by either a gas engine-operated pump of 3-m.g.d. capacity, handling the flows up to this point, or two electric-powered pumps of 3-m.g.d. and 1.5-m.g.d. capacities pumping to the detritor. From the detritor the sewage flows by gravity to the combined mixing chamber and flocculator tank installation, and finally to the 60-ft. circular type clarifiers (Figure 2).

Provisions for chlorination are available and used, both at the comminutor for odor control and on the effluent line at a point between the clarifiers and the effluent side of the control chamber. The contact time for post-chlorination is limited to the flow time for about 850 ft. of effluent line. Dollies are used for handling and transporting ton containers for the chlorine supply. Sludge is digested in a two-stage system (Figure 3), then dried on open beds before ultimate disposal.

The feeding of chemicals is accomplished by means of two 18-cu. ft. dry feeders for hydrated lime and two 300-gal. ferric chloride feeder tanks of the decanting type. These feeders are actuated by means of a transmitter mechanism attached to a venturi meter. A 250-gal. ferric chloride dissolving tank is located on the second floor of the building and the lime hoppers are charged from the same floor. The solution feed lines from both the ferric chloride decanters and the lime solution pots are carried by means of a home-made adjustable channel on a continuous downgrade to the mixing chamber. This is a change of design to eliminate air slug and sediment stoppages incurred in the original installation.

Application of Chemicals

Chemicals have been used for varying periods of time since the plant was placed in operation. The determining

* Presented at 25th Annual Meeting, Ohio Sewage and Industrial Wastes Treatment Conference; Columbus, Ohio; June 20–22, 1951.

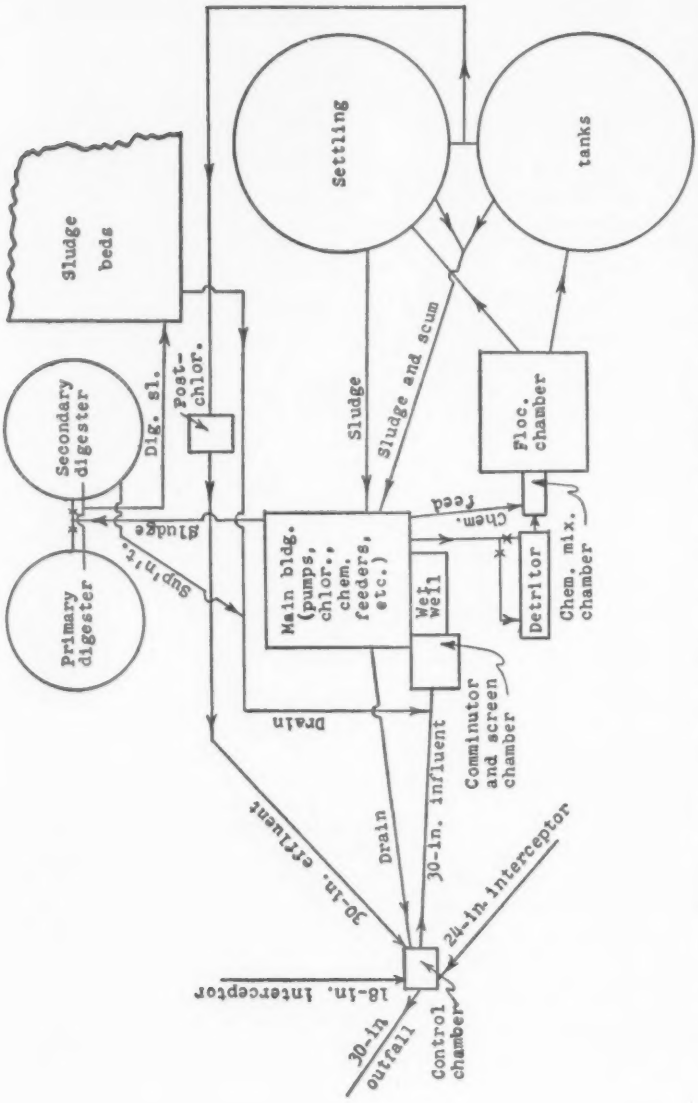

FIGURE 1.—Flow diagram of Wooster, Ohio, sewage treatment plant providing primary treatment with part-time chemical treatment and separate sludge digestion.

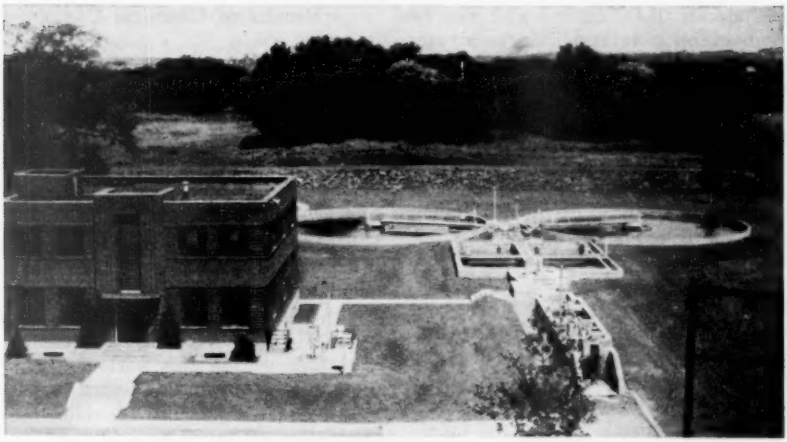

FIGURE 2.—Major primary and chemical treatment units of Wooster, Ohio, plant.

factors in using chemicals are primarily the quantity and rate of flow of the receiving stream and the D.O. content of the stream. A value of 5 p.p.m. of D.O. at the effluent discharge point has been established as a minimum; lower D.O. content requires chemical treatment. For several years it has been possible to maintain a D.O. higher than 5 p.p.m. by the use of chlorine when otherwise chemicals probably would have been required. It is standard operating procedure to start using chlorine for disinfection when the stream D.O. starts its seasonal decline due to the weather, low flow, or the effects of the effluent from the plant. This use of chlorine definitely retards

FIGURE 3.—Sludge digesters and drying beds of Wooster, Ohio, plant.

the stream D.O. decline and can be counted upon to stabilize the stream condition, other factors remaining more or less constant. A minimum of 0.5 p.p.m. residual after 10 min. is the basis of chlorine feed rate.

When chemicals are used, dosages of approximately 1 grain of ferric chloride and 10 grains of calcium oxide per gallon are required. These dosages are based upon laboratory settling and operational results. Past experience indicates that several days of adjustments of the feeding machines are required to get the proper dosages wanted. This time element is partly due to the long periods of storage of the chemical and partly due to the infrequent use of the machines themselves. After acquiring the proper adjustments the rates of feed remain relatively stable.

The chemicals have been applied in two different ways, with practically the same results and the same demand for the chemical to obtain these results. In the first method, the iron and lime is applied to the raw sewage, rapidly mixed, flocculated, and settled out in the clarifiers, with the resulting sludge being pumped to the digester. The second method used comprises primary sedimentation with the chemical being applied to the effluent, rapidly mixed, flocculated, and settled out in the secondary clarifier, with the resulting sludge being pumped to the digester. The second method requires practically the same dosage as the first method to obtain similar results. Another method of feeding the chemical, now contemplated for trial, consists of primary sedimentation with chemical being applied to the effluent of the primary clarifier, and the sludge of the secondary clarifier being pumped back constantly into the raw sewage. It is hoped that this method, after several days use, may allow sufficient decrease in the dosage applied to cut operational costs.

Results of Chemical Use

Chemicals were used 46 days during 1939, 48 days in 1940, 71 days in 1941, 25 days in 1942, 46 days in 1944, 6 days in 1945, 11 days in 1946, and 19 days in 1949, for a total of 272 days to date. During 1939 and 1940, the records (Table I) indicate increases in reduction of suspended solids of 39.6 and 24.6 per cent, respectively, by the use of chemicals. These percentages are calculated only on the period during which chemicals were used on an "on and off" basis. No other breakdown was made during this period. It is also true that during this period chemicals were used part of the time for demonstration purposes and part of the time to determine the best feeds for use at the plant.

The years 1941, 1942, 1944, and 1946 indicate an increase from 65.1 to 83.2 per cent in the reduction of suspended solids, or an increase of 27.8 per cent reduction, by the use of chemicals; similarly the B.O.D. reduction was increased from 39.8 to 62.0 per cent, or an increase of 55.8 per cent by the use of chemicals. Settleable solid reduction increased from 90.6 to 97.0 per cent during the same period. These values are based on the averages of the entire year's operation for the stated years. In the years during which chemicals were used for any length of time, the use was not on a continuous basis over the entire period, but with breaks ranging from several days to several weeks, due to relief from sufficient quantities of rainfall.

It is interesting to note that for the years 1941 and 1944, when chemicals were used for an extended period, the suspended solid averages were 27 p.p.m. (17.0 per cent) heavier in the raw sewage during this period, whereas the clarifier effluent averages dropped 27 p.p.m. (48.2 per cent). During the same periods the B.O.D. of the raw sewage increased 34 p.p.m. (21.5 per cent), although the effluent dropped 30 p.p.m. (29.8 per cent).

TABLE I.—Comparative Operating Results with and without Chemical Treatment at Wooster, Ohio, Sewage Treatment Plant

Year	Chemical Treatment			Chlorine	Operating Results					
					5-Day B.O.D.			Suspended Solids		
	Days applied / not applied	Lime[1] (grains/gal.)	$FeCl_3$ (grains/gal.)	days applied / p.p.m. appl'd	Raw (p.p.m.)	Effluent (p.p.m.)	Removal (%)	Raw (p.p.m.)	Effluent (p.p.m.)	Removal (%)
1939[2]	46	9.11	1.17	43	159	69	56.6	188	32	83.0
	224	—	—	7.31	182	110	39.6	166	51	69.3
1940	48	8.10	0.95	66	125	51	59.2	129	19	85.2
	307	—	—	4.98	140	84	40.0	136	47	65.4
1941	71	7.40	0.88	59	181	65	64.1	185	24	87.0
	294	—	—	4.56	155	98	36.8	154	51	66.9
1942	25	7.30	0.92	20	207	85	59.0	219	32	85.5
	332	—	—	4.87	159	91	42.8	163	51	68.7
1943	none	—	—	30	—	—	—	—	—	—
	350	—	—	3.95	134	94	29.9	148	59	60.1
1944	46	7.15	0.81	101	203	77	62.1	187	35	81.4
	313	—	—	4.55	161	104	35.4	164	62	62.1
1945	6	7.30	0.81	58	127	60	52.8	181	44	75.7
	340	—	—	4.20	125	72	42.4	152	60	61.5
1946	11	6.80	1.60	51	192	73	62.0	171	41	76.0
	346	—	—	5.00	150	84	44.0	145	54	62.8
1947	none	—	—	27	—	—	—	—	—	—
	340	—	—	4.40	131	81	38.2	143	55	61.5
1948	none	—	—	66	—	—	—	—	—	—
	358	—	—	5.87	154	90	41.6	167	63	62.3
1949	20	6.4	1.20	133	167	84	49.7	216	54	75.0
	341	—	—	6.31	165	98	40.6	196	67	65.8
1950	none	—	—	80	—	—	—	—	—	—
	349	—	—	6.49	144	88	38.9	143	51	64.3

[1] As CaO.
[2] Beginning in April.

Due to the normal lack of solids in the sewage during the night, it has become standard plant practice to discontinue dosage of chemicals from approximately 11 P.M. until 7 A.M. the next morning. This has materially reduced the cost, as well as the amount of chemical handled as sludge, without any apparent reduction in the operating efficiency. Chlorination of the effluent is carried out over the entire 24-hr. period.

It is recognized that the data available are scanty and that for actual comparable results of chemical treatment all samples should be run as raw and treated. However, due to the small size of the operating force, together with the additional load of summer operations during the time when chemical treatment is indicated, not much time is left to devote to work other than routine.

In conclusion, as a supplemental means of treatment the use of chemicals at Wooster has accomplished its purpose.

ARNOLD APPOINTED NPA WATER RESOURCES DIVISION DIRECTOR

Appointment of Gerald E. Arnold as director of the Water Resources Division, was announced recently by the National Production Authority, U. S. Department of Commerce. Mr. Arnold succeeds Harvey S. Howe, whose services were loaned to NPA by Lock Joint Pipe Co. to organize the Water Resources Division and head it until a permanent director could be obtained.

The new director received the George W. Fuller Memorial Award of the American Water Works Association in 1938 for "distinguished service in the water supply field." He comes to NPA from San Diego, Calif., where he served the municipality for six years, first as Assistant City Manager and later as Director of the Water Department.

Mr. Arnold's broad experience in the water resources industry includes work in construction of facilities, manufacture of components, operation of municipal plants, private irrigation and flood control operations, and supervision of governmental utility activities.

PRODUCTION OF VOLATILE NEUTRAL COMPOUNDS DURING DIGESTION OF SEWAGE SOLIDS AND INDUSTRIAL WASTES *

By H. Heukelekian and H. F. Berger

In the anaerobic digestion of sewage solids, large quantities of volatile acids are produced. The nature and quantities of these acids under various conditions of digestion have been reported previously (4). Only three of the lower volatile acids were found; namely, acetic, butyric, and proprionic. These acids serve as precursors of methane; that is, methane-producing organisms utilize these substances. It has been shown that, in addition, these organisms can utilize formic, valeric, and caproic acids. Investigations dealing with the detection of the higher fatty acids during sludge digestion are in progress. The corresponding alcohols, such as methyl, ethyl, butyl, propyl, and isopropyl, are also utilized. For a better understanding of the mechanism of digestion it is important to determine whether such alcohols are produced during the anaerobic digestion of sewage sludge and industrial wastes. No information as to their occurrence is available. This paper deals with the detection of the alcohols and acetone and acetaldehyde produced in the course of digestion. These substances are collectively referred to in this paper as "volatile neutrals" to differentiate them from volatile acids.

Methods and Procedure

It is necessary first to separate the volatile neutrals from the sludge, then to separate them from the volatile acids, and finally to identify the specific groups of volatile neutral compounds. The volatile neutrals and volatile acids were separated from the sludge sample by the filtration method developed by Heukelekian and Kaplovsky (3) for the determination of volatile acids. The filtrate was adjusted to pH 11.5 and distilled. The distillate contained only the volatile neutrals, as the volatile acids were converted to the sodium salts, which are not volatile. The selection of a method for the determination of alcohols was based on an expected alcohol concentration of less than 1,000 p.p.m. The method used by Stahly, Osburn, and Werkman (6) was selected. This method is based on the oxidation of the alcohols with potassium dichromate to the respective volatile acids, with subsequent distillation of the volatile acids formed. The identity of the alcohols was determined by subjecting the distillate to Werkman's partition method for the identification of lower acids. This could only be done if the alcohols were present in sufficient concentration to give a volatile acid concentration of more than 0.02 *N*, otherwise only the total quantity of alcohol could be determined.

Acetone was determined in an aliquot from the first distillation, before oxidation, by Goodwin's (2) modification of Messinger's method based on iodoform formation. When both acetaldehyde and acetone are present the iodine equivalent of the former is subtracted from the total to obtain the value for acetone. Acetaldehyde was determined in the unoxidized aliquot by the method of the Association of Official Agricultural Chemists (1).

To check their suitability and accuracy the methods used were sub-

* Paper of the Journal Series, New Jersey Agricutural Experiment Station, Rutgers University, New Brunswick, N. J.

jected to a recovery test by the addition of 1,000 p.p.m. each of ethanol, isopropanol, and n-butanol. The recoveries were better than 90 per cent for ethanol and n-butanol, but only 65 per cent for isoproponal, because of the incomplete oxidation of the latter with dichromate. Accordingly, if significant quantities of isoproponal are present, the concentration of this alcohol can be multiplied by a factor of 100 to 65. Other known mixtures containing acetone and acetaldehyde, in addition to volatile alcohols, were analyzed by the above method. During the oxidation acetone remains unchanged but the aldehyde is oxidized to acetic acid. The quantity of acetic acid equivalent to the acetaldehyde must be subtracted from the total in order to determine the alcohols; the acetone originally present must also be subtracted from the total after oxidation, and the difference is that derived from isoproponal.

The methods outlined above were followed in determining the volatile neutrals in digesting unseeded sewage solids with and without lime additions and in seeded mixtures of various volatile matter ratios. Corollary determinations of volatile acids, daily gas measurements, composition of the gas, and solids and ash determinations were made. Similar studies were made on the digestion of a number of industrial wastes. The digestion temperature was 20° C.

TABLE I.—Production of Volatile Acids and Volatile Neutrals During Sewage Sludge Digestion; Unseeded Fresh Solids

Digestion Time (days)	pH	Tot. Vol. Acids (p.p.m.)	Tot. Alcohols, as Ethanol (p.p.m.)	Acetaldehyde[1]	Acetone[1]
0	6.10	—	—	0	0
5	5.70	1,094	11	0	0
14	5.21	1,686	14	+	0
21	5.31	1,261	21	0	0
28	5.37	1,981	21	0	+
35	5.51	2,352	17	+	+
42	5.58	2,293	24	+	0
49	6.05	1,758	18	++	0
56	6.78	1,607	21	+	0
63	6.68	1,091	13	+	0
70	6.80	1,085	19	0	0

[1] + = approximately 10 p.p.m.; ++ = 10 to 30 p.p.m.; 0 = absent.

TABLE II.—Production of Volatile Acids and Volatile Neutrals During Sewage Sludge Digestion; Seeded 2:1

Digestion Time (days)	pH	Tot. Vol. Acids (p.p.m.)	Tot. Alcohols, as Ethanol (p.p.m.)	Acetaldehyde[1]	Acetone[1]
0	6.57	28	42	0	0
5	6.70	234	15	0	0
8	6.70	146	—	0	0
14	6.31	73	14	0	0
21	6.96	103	12	+	0
28	6.92	79	6	+	0

[1] + = approximately 10 p.p.m.; 0 = absent.

Results

The results from the digestion of unseeded fresh solids are given in Table I. During the 70-day period, the total alcohols fluctuated between 11 and 24 p.p.m., and acetaldehyde and acetone were present in even smaller concentrations. Similar results were obtained with limed fresh solids (results not included). The results from the digestion of seeded mixture (2 parts of raw volatile solids to 1 part of digested volatile solids) are given in Table II. Again, the concentration of the alcohols, acetaldehyde, and acetone are low. Similar results were obtained in the digestion of 4:1 mixture. It is apparent that the predominant intermediate compounds formed in the digestion of sewage solids are volatile acids, with only traces of volatile neutral compounds. It does not seem that the low level of the volatile neutrals in the course of sludge digestion can be attributed to the rapid conversion of these compounds to methane, because in the more slowly digesting unseeded sludge rapid gas production did not take place and the volatile neutrals, if produced, should have ac-

cumulated. The absence of the volatile neutrals in appreciable concentrations indicates that liquefaction results not in the formation of volatile neutrals, but of volatile acids. A possible explanation for this phenomenon is given hereinafter.

The production of volatile neutrals during the digestion of concentrated white water from a paperboard mill was studied in a mixture seeded 1:1 on the basis of volatile matter. Nitrogen was added in the form of ammonium carbonate to make up for the

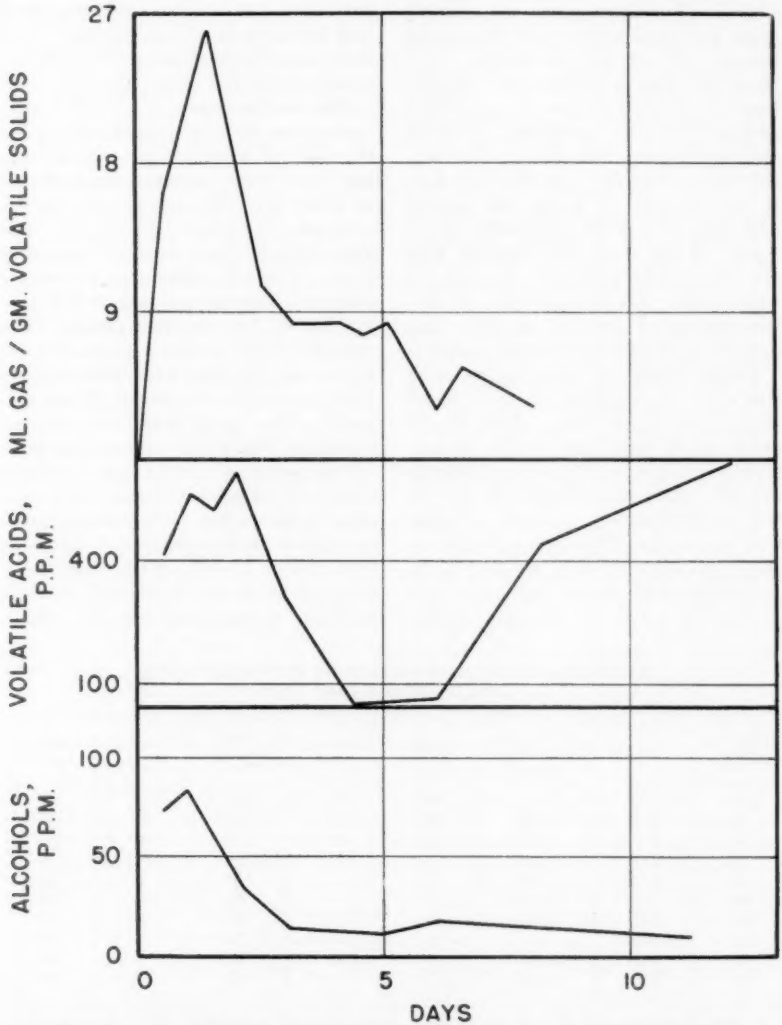

FIGURE 1.—Gas, volatile acids, and total alcohol production during the digestion of seeded mixture of concentrated white water.

nitrogen deficiency of the waste. The gas produced and the volatile acid and volatile alcohol contents are plotted in Figure 1. The peak of gas production was reached in three days, during which period the volatile acids concentrations were also at a high level. The gas production and volatile acids decreased thereafter, but the volatile acids increased again without a simultaneous rise in gas production. The secondary rise in volatile acid production may be attributed to the liquefaction of more resistant materials than during the first peak. The alcohol content was 76 p.p.m. the first day; it increased to 88 p.p.m. the second day and decreased gradually to 19 p.p.m. by the sixth day, parallel with the decrease in volatile acids and gas production. No acetaldehyde or acetone could be detected at any time during the digestion of white water.

Three series of experiments were run with the digestion of spent broth from yeast production. Two of the experiments were run under deliberately overloaded conditions in order to increase the concentration of the intermediate compounds, such as alcohols and acids. The seeding ratios in these two mixtures were 6 and 9 parts of volatile solids in the waste per part of volatile solids in the ripe sludge. The ripe sludge was acclimatized before the experiment by gradually increasing additions of the waste.

The alcohol level in the 1:20 mixture was very low and acetaldehyde and acetone were absent during the entire period of digestion. The gas production in this mixture reached a peak during the first day and decreased to a low level on the second day. For these reasons the results from this experiment are not reported.

The results from 6:1 and 9:1 mixtures were similar to each other; for the sake of brevity, the results from only the former mixture are included in Table III. The gas production figures are not included because the volumes were low and comparatively constant. The pH value was 4.4 at the start and increased slowly to 6.9 during the 89-day digestion period. The volatile acids reached a peak of 4,490 p.p.m. in 23 days and decreased to 1,505 p.p.m. by the end of the experiment. The spent broth as received contained 356 p.p.m. of total alcohols, which increased to 428 p.p.m. during 6 days of digestion and decreased sharply thereafter. The concentration of alcohols during the first 6 days was high enough to make the successful determination of the individual alcohols possible. It was found that the original

TABLE III.—Production of Volatile Acids and Volatile Neutrals During Digestion of Yeast Waste

Digestion Time (days)	pH	Tot. Vol. Acids, as Propionic (p.p.m.)	Ethanol[1] (p.p.m.)	Butanol[1] (p.p.m.)	Total Alcohols[1] (p.p.m.)	Acetaldehyde (p.p.m.)	Acetone (p.p.m.)
0[2]	4.40	—	332	24	356	30	25
2	4.90	1,190	377	15	392	20	20
6	5.10	1,860	401	27	428	10	20
12	4.80	4,090	—	—	166	10	20
16	4.55	4,220	—	—	87	5	25
23	4.60	4,490	—	—	87	0	25
30	5.12	4,175	—	—	76	0	25
37	5.00	—	—	—	78	0	20
55	6.00	3,180	—	—	59	0	20
89	6.90	1,505	—	—	47	0	20

[1] Total alcohol concentration too low, from 12th day on, to determine individual alcohols; total alcohols reported as p.p.m. ethanol.

[2] Waste as received; not seeded.

TABLE IV.—Sludge Analyses and Production of Volatile Neutrals During Digestion

Digestion Time (days)	pH	Tot. Alcohols, as Ethanol (p.p.m.)	Acetaldehyde[1]	Acetone[1]
0	7.50	112	0	0
1	6.25	114	0	0
2	5.95	65	+	0
3	5.88	107	+	0
5	5.95	53	+	0
7	6.15	—	—	—
12	6.30	7	+	0

[1] + = approximately 10 p.p.m.; 0 = absent.

broth contained 332 p.p.m. of ethanol and 24 p.p.m. of butanol. Ethanol increased to 401 p.p.m. in 6 days, while there was no significant change in the butanol concentration. The original waste also contained 30 p.p.m. of acetaldehyde and 25 p.p.m. of acetone. There was no increase in the concentration of these two compounds during digestion, but acetaldehyde decreased and completely disappeared, whereas there was no significant change in the acetone concentration.

Waste from the production of candied fruit was selected because of its high sugar content and consequent possibility of the production of alcohols upon digestion. This waste is deficient in nitrogen, which was corrected by the addition of ammonium hydroxide. Previously acclimatized ripe sludge was used. The seeding ratio was 1 part of volatile solids in the waste to 5 parts of volatile solids in the ripe sludge. Gas production reached a peak in 2 days and decreased rapidly thereafter. Alcohol was present in comparatively high concentration initially and decreased during digestion to a negligible value (Table IV). A positive acetaldehyde reaction was obtained at times, whereas acetone was absent during the entire period.

Discussion

The quantities of alcohol and acetone produced in the anaerobic digestion of sewage solids were found to be low. In the digestion of high carbohydrate wastes, such as white water, spent yeast broth, and candied fruit wastes, somewhat higher quantities of these compounds were produced. In contrast, volatile acids are produced in large quantities from all these wastes as well as from sewage solids. There are two possible explanations for these phenomena: (1) the hydrolysis of complex organic compounds proceeds only by way of organic acids and (2) organic acids as well as alcohols and acetone are produced by hydrolysis, but the rate of destruction of the alcohols and acetone is greater than that of organic acids and, therefore, the former compounds do not accumulate, while the organic acids, because of their unfavorable effect on the hydrogen ion concentration, are not utilized by methane organisms as fast as they are produced. If, on the other hand, alcohols and acetone are produced only in small quantities, it may be because the organisms responsible for bringing about the transformation of complex organic materials to these intermediate stages either are not found in ripe sludge or do not find the proper conditions for their development. In view of the fact that all the various organisms necessary to perform all the biochemical reactions utilized in the different treatment processes are found in sewage and wastes, it would be surprising if a few of the organisms capable of producing alcohols from complex organic compounds were not present in sewage. One of the important organisms utilized industrially for the production of acetone and n-butyl alcohol is *Clostridium acetobutylicum*. By mass inoculation of sterile fresh solids with this organism and determination of the quantities of alcohol and acetone produced, it should be possible to answer some of the questions raised. In the absence of any other organism, the alcohols and acetone, if produced, should accumulate. On the other hand, if they are not pro-

TABLE V.—Production of Volatile Acids and Volatile Neutrals During Fermentation of Sterile Fresh Solids by *Cl. acetobutylicum*

Digestion Time (days)	pH	Tot. Vol. Acids, as Propionic (p.p.m.)	Tot. Alcohols, as Butanol (p.p.m.)	Acetaldehyde	Acetone (p.p.m.)
0	5.10	2,150	121	0	24
2	5.12	1,955	184	0	44
3	5.10	2,385	127	0	53
7	5.10	2,465	114	0	54
9	5.20	2,235	127	0	104

duced it should be safe to conclude that it is not the absence of the organisms that is responsible for the lack of production of alcohols and acetone from sludge and industrial wastes, but the improper environmental conditions that prevent the organism from developing.

Such an experiment was performed. Sterile fresh solids were inoculated heavily with a pure culture of *Clostridium acetobutylicum*. The results are given in Table V. It is apparent that vigorous production of organic acids, alcohols, acetaldehyde and acetone was not obtained. Peterson and Fred (5) obtained the following quantities of solvents from the fermentation of 6 per cent corn mash by the organisms in 75 hr.:

Acetone	4,200 p.p.m.
Butanol	7,000 p.p.m.
Ethanol	1,800 p.p.m.

It appears, therefore, that the failure of production of alcohols and acetone in the anaerobic digestion of sewage sludge and industrial wastes is due neither to the absence of the necessary organisms nor to the rapid utilization of these products by other organisms, but rather to the absence of special conditions required for the development of these organisms. The low pH values under which this experiment was conducted were not responsible for the small quantities of alcohols and acetone produced, because, according to Peterson and Fred (5), solvent production becomes rapid at the time of maximum acidity. Hence, some other special condition is responsible for the lack of vigorous alcohol and acetone production by these organisms from sewage sludge and industrial wastes. It is known that *Clostridium acetobutylicum* ferments corn mash and glucose vigorously. It appears, therefore, that the food in the sewage sludge and the industrial wastes used is not suitable for the development of this organism and that other acid producing types find the conditions more favorable for their development.

The unsuitability of food for alcohol and acetone production may be related to either the type or the concentration of the food. Carbohydrates, such as corn mash and sugars, have been used for the production of alcohols and acetone by *Clostridium acetobutylicum*. Fresh solids contain little if any such available carbohydrates. The type of carbohydrate in the white water used in this study is cellulose, which is not available for this type of fermentation. However, in spent yeast broth and candied fruit wastes, sugars are present. The failure of these wastes to produce large quantities of alcohol and acetone might be attributed to the relatively low concentration of these available carbohydrates. In the industrial production of butyl alcohol and acetone 6 per cent corn mash is used; laboratory culture media for this organism utilize high concentrations of sugars.

The possibility of recovery of alcohols and acetone by directing the course of anaerobic digestion of sewage sludge and industrial wastes for the production of these intermediate products seems rather remote.

Summary and Conclusions

To gain an added understanding of the process of anaerobic digestion with a view toward regulating conditions favoring the most desirable course and recovering certain by-products, volatile neutral intermediates were isolated from digestion mixtures and quantitatively determined.

The following wastes were digested anaerobically in the laboratory and analyzed for alcohols, acetone, and acetaldehyde:

1. Sewage sludge, unseeded and seeded in various ratios, limed daily to maintain pH 7.0.
2. White water from a paperboard mill.
3. Spent nutrient from a compressed yeast plant, seeded in various ratios.
4. Sugar waste from the manufacture of candied fruit.

From the studies reported, the following general conclusions were drawn:

1. Three types of high carbohydrate industrial wastes proved poor producers of volatile neutrals, alcohols, acetone, and acetaldehyde.
2. Sterile sewage sludge exposed to the action of *Cl. acetobutylicum*, a known vigorous producer of alcohols and acetone, produced volatile neutrals in very low concentrations.
3. Sewage sludge provides an unsuitable substrate for microorganisms which produce volatile neutrals.
4. Recovery of commercially important organic solvents from sewage sludge would be impractical, if not impossible, with the present knowledge.
5. Anaerobic digestion of sewage sludge and industrial waste proceeds normally by way of intermediate production of lower volatile acids, which are then utilized by methane bacteria.

Acknowledgment

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Industrial Wastes

BIO-ASSAY METHODS FOR THE EVALUATION OF ACUTE TOXICITY OF INDUSTRIAL WASTES TO FISH

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Committee on Research, Subcommittee on Toxicity, Section III, Federation of Sewage and Industrial Wastes Associations

I. INTRODUCTION

The bio-assay procedures described herein are intended for use in industrial and other laboratories in evaluating the toxicity of wastes and of other water pollutants to fish. These practical tests can be used to determine whether or not a waste or waste component is markedly toxic; if it proves decidedly toxic, they can be used to determine also the degree of toxicity. On the basis of toxicity determinations, one can judge whether or not a waste can be discharged at a given rate without causing direct injury to fish and other organisms in the receiving water. If damage to fish life already has been observed, one can judge whether it is attributable to the toxicity of a particular waste, waste component, or combination of wastes, or is ascribable to other causes. When it has been determined that the discharge of a certain waste has been injurious or would be dangerous to valuable aquatic life, treatment of that waste or some other method of safe disposal should, of course, be devised. It is not the purpose of this report to consider or suggest methods of disposal and treatment of toxic wastes. However, the data obtained through the use of the recommended bio-assay procedures can be very helpful in deciding how much treatment is required and what methods are appropriate. The degrees of treatment and dilution necessary obviously depend on the degree of toxicity. Finally, the bio-assay procedures can be used to test the effectiveness of any treatment provided.

The value of such biological tests of waste quality, in connection with the control of waste disposal and treatment, has been increasingly realized and has been discussed very recently elsewhere (2)(7). Chemical examina-

[1] Representing the U. S. Public Health Service (Cincinnati, Ohio), the West Virginia University, the New York State Conservation Department, the U. S. Fish and Wildlife Service, the Atlantic Refining Company, the Academy of Natural Sciences of Philadelphia, the Foundation of Applied Research, the U. S. Fish and Wildlife Service (formerly; now with the U. S. Public Health Service), and the Institute of Paper Chemistry, respectively. The interest and helpful advice of Dr. W. Rudolfs, chairman of the Research Committee, the consultants named below, and many others who lent assistance and encouragement in the course of the preparation of this report, are gratefully acknowledged. Consultants who participated in discussions throughout one or more committee meetings or reviewed progress reports and submitted critical comments were: J. B. Anderson, E. F. Eldridge, J. Greenbank, C. Henderson, C. L. Hubbs, M. Katz, C. B. Kelly, S. F. Snieszko, C. M. Tarzwell, and J. S. Wiley.

tion of complex industrial wastes alone usually does not yield sufficient information. Many of their various toxic components cannot be readily detected, separated, and measured by chemical means. Moreover, the degree of toxicity of every one of these numerous substances and different mixtures of chemicals which occur in industrial effluents is not known. The toxicity of wastes can be greatly influenced by interactions between their individual components and the dissolved minerals present in widely varying amounts in receiving waters. Furthermore, different kinds of fish are not equally susceptible to toxic substances, and much of the pertinent published information is based on experiments with hardy species only. Therefore, the toxicity of an industrial waste to local fishes in their natural medium must be detected and evaluated directly, through biological tests under appropriate experimental conditions.

The chief purpose of the present recommendations is the establishment of a sound practical criterion by which the probability of direct injury to fish life, due to acute toxic action of the pollutants tested, can be judged. The recommended tests show what concentrations, or dilutions, of a toxic substance or effluent are fatal to some fish within a certain test period. The duration of the exposure is necessarily limited. Knowledge of the relative acute toxicity of a waste is very useful in the estimation of a safe disposal rate. However, conclusive evidence that pollution has been entirely harmless to fish life after an extended period of time can be obtained only through careful biological study of the receiving body of water.

It is convenient, for present purposes, to regard as toxic action any direct lethal action of pollutants, including both internal and external action. Indirect effects of pollution, such as mortality of fish caused by depletion of dissolved oxygen through chemical or biological oxidation of waste components, also are important. However, death due to deficiency of dissolved oxygen in polluted water should be distinguished from death due to toxicity. In order to detect and to evaluate the direct lethality of wastes only, adequate dissolved oxygen concentrations must be maintained during toxicity tests.

Reasonable uniformity of experimental procedure and of the manner of presentation of bio-assay results is essential. Without such standardization of method there can be little progress in the refinement of the application of test results to practical waste disposal problems. Widespread adoption of uniform methods will promote the gradual accumulation of much-needed comparable data and effective pooling and sharing of pertinent practical experience by all interested workers.

However, rigid standardization of experimental material and conditions is not desirable, for it would tend to defeat the purpose of practical tests. Strict comparability of test results could be achieved only by sacrificing much of their relevance and applicability to practical problems in different localities. Experimental water (that is, water used as a diluent) and test animals which best suit the purpose of each bio-assay should be selected. With respect to other features, the tests can be more or less uniform.

It is believed that the standardization of experimental conditions and procedures recommended herein will ensure adequate uniformity, reproducibility, and general usefulness of the bio-assay results, without interfering unduly with the adaptability of the tests to different local circumstances and problems. Other parallel tests performed under experimental

conditions more closely resembling the actual environmental conditions in a given body of water (with respect to fluctuations of temperature and dissolved oxygen, and so forth) sometimes can be highly instructive. Occasionally they may reveal harmful effects of a waste which are not demonstrable by the standardized tests described. Duplication of all variable conditions which occur in natural and polluted fish habitats is seldom possible and necessary, however.

The present recommendations are based on the experience of many investigators. Full discussion of the many practical and theoretical considerations involved would be out of place in this concise report. Much helpful, detailed information of explanatory value can be found in the literature.[2]

Because of the economic and recreational importance of many fish species directly affected by pollution, and for other reasons, fishes already have been very often used and recommended as test subjects. Further justification of their use for present purposes evidently would be superfluous. The value of other aquatic organisms, including those which are important as fish food, without which fish populations cannot thrive, or as agents of natural purification of polluted waters, should not be overlooked, however. Although many fishes are very susceptible to most poisons, some of the lower organisms are killed by concentrations of certain pollutants which may be harmless to fish. Therefore, the development of standardized toxicity bioassay methods involving the use of experimental material other than fish (for example, microcrustacea, marine shellfish, etc.) also is planned.

The recommendations which follow are presented in two text sections and three appendices. Section II deals with the simplest routine procedure, which has been found to be widely applicable. This basic procedure is suitable for the detection and evaluation of acute toxicity which is not associated with excessive oxygen demand and is due to substances which are relatively stable and are not extremely volatile. Excessive oxygen depletion, and also gradual detoxification or reduction in concentration of any important components of the wastes or solutions tested, tend to interfere, more or less, with accurate estimation of toxicity by the routine method. In Section III, some more laborious, auxiliary methods are described, by which such difficulties can be overcome, or the reliability of test results can be enhanced. Some suggestions concerning toxicity tests which are more prolonged than those recommended for general routine application are included. It should be understood, however, that details of methods for the detection and study of chronic toxicity are not within the scope of the present recommendations. Chronic or cumulative intoxication can interfere seriously with the growth and reproduction of organisms, or increase their susceptibility to disease, without killing them outright, and its effects sometimes become apparent only after a very long time.

[2] The most pertinent published treatise is a comprehensive manual by Hart, Doudoroff, and Greenbank (5), which is out of print, however, having been prepared for limited distribution only. Most of the simplified and more concise recommendations presented herein derive directly from the recommendations made by these authors. Somewhat different, and less detailed and specific, but more or less parallel recommendations were published at about the same time by Ellis, Westfall, and Ellis (4). A comprehensive critical review of literature on the toxicity of industrial wastes to fish has been undertaken recently. References to earlier reviews and summaries, as well as numerous original sources of information, can be found in the first part of the review by Doudoroff and Katz (3).

II. OUTLINE OF ROUTINE BIO-ASSAY PROCEDURE

A. Choice and Preparation of Test Animals

1. Kinds of Fish to Be Used as Test Animals

a) Species selected should not be very resistant to adverse water quality conditions, but should withstand captivity well.

b) They should be species which are common in unpolluted portions of the body of water receiving the waste to be tested, or at least species which are known to inhabit similar waters in the same major watershed. If available, species which are deemed important locally should be given preference.

c) Although any fish species which suits the purpose of the investigation may be used, species belonging to any of the following widely distributed and important families are particularly recommended, and should be selected unless there is good reason for making a different choice:

Centrarchidae (sunfishes, basses, crappies).
Salmonidae (trouts, charrs, salmons).
Cyprinidae (true minnows), exclusive of carp and goldfish.
Catostomidae (suckers).

2. Identification of Test Animals

The fish used for each individual toxicity evaluation must all be of the same species. They should be identified at least to genus, and preferably to species. The correct scientific name should be stated when the test results are reported.[3]

3. Source of Test Animals

The fish are to be obtained from any single common source (stream, lake, hatchery, etc.), but preferably from the body of water receiving the pollutant tested. They should all be collected and brought to the laboratory at about the same time.

4. Size of Test Animals

a) The length of the largest fish used in an individual bio-assay should not be more than 1.5 times the length of the smallest specimen used.

b) Small specimens, averaging less than 3 in. in length, generally are the most convenient and desirable test animals.

5. Holding Test Animals

Stocks of fish for test purposes may be kept initially in any suitable enclosures or containers (for example, small ponds, live-boxes, screened pens, concrete or wooden tanks, glass aquaria, etc.), and in any water of suitable quality and temperature, in which they remain in good condition until they are needed. The care and feeding of test animals are discussed in Appendix I.

6. Acclimatization of Test Animals

a) The test animals should be acclimatized for at least one week (preferably 10 days or longer) to conditions similar to those under which the tests are to be performed, with regard especially to the temperature and the chemical properties (or source) of the water.

b) The fish should be fed at fairly regular intervals at least three times a week, and preferably daily, during the acclimatization period, but should not be fed for a period of about two days before they are used in a test.

7. Fitness of Test Animals

a) The percentage of specimens dying or becoming seriously diseased in the acclimatizing aquarium during a period of four days immediately preceding a test must be less than 10 per

[3] A recent publication of the American Fisheries Society (1) is a helpful guide to proper widely-approved nomenclature of fishes.

cent. Otherwise, the test animal lot should be deemed unfit for use until the incidence of disease and the mortality rate decline sufficiently.

b) Specimens used for test purposes must show no evident symptoms of disease, or abnormalities of appearance or behavior, at the time of their transfer to test containers (that is, experimental aquaria).

B. Selection and Preparation of the Experimental Water (Diluent)

1. Usual Source of Experimental Water

a) The experimental water which is to be used as a diluent and acclimatizing medium should be obtained from the body of water which receives the waste under investigation.

b) When the toxicity of a waste alone is to be determined, the water should be obtained at a point where there is no pollution or contamination with wastes from any source.

c) When a stream water receiving the waste to be tested is subject to previous contamination with other wastes, the toxicity of that waste in conjunction with the contaminants already present in the receiving water also must be considered in judging safe rates of discharge. For the evaluation of this toxicity, it is necessary to use experimental water obtained immediately upstream from the point of discharge of the waste to be tested, but outside the zone of its influence. Such an evaluation is impossible when the test animals cannot be held successfully in the diluent water, because of previous contamination with injurious substances.

2. Substituted Water

If the uncontaminated experimental water recommended above (item B-*1*-b) cannot be supplied from the body of water under consideration, water of similar quality, with respect to its dissolved mineral content, should be obtained from some other source, or prepared. Such water can be prepared by adding appropriate chemicals to some available natural water of suitable quality, which may first be diluted, if necessary, with distilled or demineralized water of assured purity. As a general rule, the calcium, magnesium, sulfate, and total dissolved solids content of this substituted water, and especially its total alkalinity, should not differ from those of the water receiving the waste tested (preferably the average values, if known) by much more than 25 per cent.

3. Modification of Water for Use as a Diluent

a) If an uncontaminated water which is to be used as a diluent contains much non-colloidal suspended material (including plankton), this should be mostly removed by sedimentation or filtration.

b) The diluent water should be well aerated with dispersed compressed air after adjusting its temperature.

c) When contaminated water is to be used as a diluent (item B-*1*-c above), unnecessary delay of its use and treatment which can result in serious alteration or removal of the pollutants present should be avoided.

C. Other Prescribed Experimental Conditions

1. Test Temperatures

a) It is recommended that the tests be performed at a uniform temperature between 68° and 77° F. (20° and 25° C.) when so-called warm-water fishes (for example, *Centrarchidae*, most *Cyprinidae*) are used as test animals, or between 54° and 64° F. (12° and 18° C.) when cold-water fishes (for example, *Salmonidae*) are used.

b) It is desirable to hold the temperature at all times within a range extending about 2° F. (or 1° C.) above and below the average test temperature selected and reported. This can be accomplished by performing the experiments in a constant-tempera-

ture room, or immersing the test containers in a large constant-temperature bath.

2. Test Containers

The containers or aquaria in which the tests are performed should be of glass. They may be cylindrical glass jars or rectangular glass aquaria with metal frames. Jars 12 in. in diameter and 12 in. high have proved very satisfactory for most tests, but larger aquaria (for example, 10- to 15-gal. tanks) sometimes are also useful. It is essential that the containers be chemically clean.

3. Depth of Liquid in Test Containers

The average depth of the liquid should be uniform in all parallel tests and should never be less than 6 in. The purpose of the latter restriction is to limit the rate of escape of any gaseous or other volatile components, which varies with the ratio of the exposed surface area to the volume of the liquid.

4. Dissolved Oxygen Content of Liquids Tested

The dissolved oxygen content of test solutions, or dilutions, should not fall at any time below 4 p.p.m. when warm-water fishes are used as test animals, and 5 p.p.m. when cold-water fishes are used. The dissolved oxygen content also should not exceed the saturation value at the experimental temperature.

5. Aeration and Oxygenation of Liquids Tested

Reaeration of the liquid in a test container occurs at the still surface exposed to the atmosphere. Sometimes the rate of absorption of oxygen from the air is not great enough to maintain the prescribed dissolved oxygen concentration. When this impediment cannot be readily overcome without resorting to artificial oxygenation (for example, by reducing the number of test animals in each test container), special methods for regulating the oxygen content, described in Section III, may be employed. Unregulated artificial aeration of waste samples and dilutions, by means of dispersed compressed air or otherwise, by which any gaseous or volatile components may be driven out of solution rapidly is not permissible, unless such removable components are known to be absent. Thorough aeration is approved treatment of some wastes before discharge, but the toxicity of a waste or waste dilution which has been aerated is often less than that of an untreated sample.

6. Number of Test Animals

a) At least 10 test animals should be used for testing each experimental concentration of the substance or waste under investigation, if final conclusions are to be based on the outcome of the test. These animals may be equally divided among two or more test containers with solutions, or waste dilutions, of the same concentration.

b) Fewer than 10 test animals may be used, however, in preliminary tests performed in order to determine what critical concentrations should be tested with the larger number of fish.

7. Total Weight and Number of Test Animals in a Test Container

a) The weight of all fish in a test container should not exceed 2 grams per liter of the liquid medium tested. Preferably, it should be about 1 gram, or less, per liter of liquid, especially when the average weight of the fish is not much more than 2 grams apiece. Sometimes the weight of fish which can be held successfully in a given volume of liquid may be further restricted by the available oxygen supply and related requirements.

b) Even when the fish used are very small, there should not be more than one fish for each liter of liquid in a test container.

D. Experimental Procedure

1. Preparation of Experimental Dilutions of Wastes

a) All dilutions required for a single toxicity evaluation should be prepared with the same sample of waste, portions of which may be stored until needed at a temperature of 32° to 39° F. (0° to 4° C.) in completely filled, tightly stoppered bottles. The duration of such storage of a sample should be held to a minimum and should be reported, along with the source and nature of the sample.[4]

b) Any undissolved material present in a sample of waste water should be uniformly dispersed, by agitation, before a measured portion of the sample is withdrawn and mixed well with a measured quantity of diluent water. However, all unnecessary exposure of the samples and dilutions to the atmosphere (aeration), through violent agitation or otherwise, should be carefully avoided.

2. Concentrations of Solutions and Dilutions Tested

a) Concentrations of dilutions of liquid industrial effluents (that is, aqueous solutions, suspensions, and emulsions of complex or unknown composition) should be expressed as per cent by volume (for example, a 10 per cent dilution equals one part of waste water in nine parts of diluent water). Concentrations of solutions of non-aqueous wastes and individual chemicals (solids, liquids, or gases) should be expressed as parts per million by weight of the substance added to the experimental water.[5]

b) An industrial effluent or a solution of uncertain toxicity may be tested first without any dilution only if it has an adequate dissolved oxygen content. If it is found to be deficient in dissolved oxygen, it must first be diluted with well-aerated experimental water until the required persistent dissolved oxygen content is just attained. The resulting concentration is the highest that can be properly tested by the routine method, but a more concentrated dilution can be prepared for testing by an auxiliary method described in Section III(B). The conduct of the test is detailed further.

c) If all or most of the test animals die, while the dissolved oxygen content of the liquid tested remains adequate, measurable toxicity of the pollutant is indicated. Progressively lower concentrations then should be tested, in order to locate the *critical concentration range*. This critical range is the interval between the highest concentration at which all test animals survive for the duration of the tests undertaken (48 or 96 hr.), and the lowest concentration at which all test animals die within 24 hr.[6] It can be roughly located through preliminary trials, with a few test animals, of a series of widely different concentrations (for example, 10 per cent, 1.0 per cent, 0.1 per cent, etc.) It can then be further delimited by testing likewise some appropriate intermediate concentrations.

[4] Extensive damage to the aquatic life of a receiving stream can result from brief, intermittent discharges of a highly toxic waste, even if the toxicity of the effluent at other times and its average toxicity are negligible. Therefore, when there are indications of considerable variations of the composition of an effluent, or when constancy of the composition is uncertain, it is necessary to test a number of fresh individual (grab) samples taken at times when the effluent is likely to be most toxic, in order to determine the maximal toxicity. A composite sample of an industrial effluent, consisting of portions collected at regular intervals of time, can be useful only when the average toxicity is to be evaluated.

[5] The inclusion of the weight of any water of crystallization as part of the weight of a hydrated, crystalline solute should be clearly indicated.

[6] The critical range also can be the interval between the former concentration and the highest concentration which can be tested successfully, when all the test animals do not die at this highest concentration within 24 hours.

d) When the critical concentration range has been approximately determined, the toxicity of the pollutant can be evaluated by testing carefully several concentrations near and within the apparent limits of this range. A series of at least four or five such concentrations should be tested. Tables of suggested test concentrations are presented and explained in Appendix II.

3. Control Tests

Along with each test of any concentration of the substance assayed, or with each series of tests of different concentrations tested simultaneously, a concurrent control test should be performed. It should be performed in exactly the same manner as the other tests, but using the diluent water alone as the medium in which the fish (controls) are held. The dissolved oxygen content of the water in the control test containers is subject to the restrictions applicable to other tests. There should be no more than 10 per cent mortality among the controls during the course of a test, and at least 90 per cent must remain apparently in good health. Otherwise, the test results cannot be deemed reliable.

4. Transfer of Test Animals

a) Test animals should be transferred from the acclimatizing aquarium to the test containers within 30 min. after the preparation of the experimental solutions or waste dilutions. This time interval should be uniform and should be recorded.

b) Test animals should be transferred from one container to another only with small-meshed dip-nets of soft material, or with wet hands. They should not be allowed to rest on any dry surface, or be held out of water any longer than necessary.

c) Any specimen which has been accidentally dropped or otherwise mishandled during transfer should be rejected and not used for test purposes until its health and freedom from injury have been established. All the test animals should be selected and graded according to size in advance, so as to avoid any unnecessary handling just before the test.

5. Duration of Tests

a) The duration of all tests should be at least 48 hr. However, tests should be continued for 96 hr. whenever this can be done conveniently.

b) In the event that more than half of the test animals survive for 48 hr. at the highest concentration that can be tested properly, the test must be continued for 96 hr. The substance or waste under investigation may be reported not to have acute toxicity which is demonstrable and measurable by the routine procedure described here only when it has been shown that most of the test animals survive for 96 hr. at this maximal test concentration. However, if toxicity cannot be demonstrated or evaluated by the routine procedure, one may have to resort to one or more of the auxiliary methods (Section III) before concluding that no significant acute toxicity can be detected and measured.

6. Observations during Tests

a) The number of fish which have died in each test container should be observed and recorded exactly 24 hr. and 48 hr. after their introduction, and also after 96 hr. if the test is continued beyond the minimum (48-hr.) test period. The numbers of fish which are alive but showing pronounced symptoms of intoxication and distress, such as loss of equilibrium and other markedly abnormal behavior, also should be noted and recorded.

b) Test animals should be deemed "dead" at the time of observation only if respiratory and other movements, either spontaneous or in response to mild mechanical stimulation (such as prodding of the animal or pressing its tail with a glass rod),

cannot be readily detected during an observation period of about 5 min. Dead fish should be removed as soon as they are observed.

c) Minimum required quantities (samples) of the liquids tested should be removed from the test containers as often as necessary for determination of dissolved oxygen, or other instructive chemical tests. In order to maintain the liquid surface at a uniform level, such samples may be replaced with equal volumes of similar solutions, or dilutions, which may be prepared at the same time as those tested and stored until needed in separate containers.

7. Feeding of Test Animals during Tests

The fish should not be fed during the tests of limited duration (96 hr. or less).

8. Expression and Evaluation of Toxicity

a) The recommended measure or index of relative toxicity is the *median tolerance limit* (symbol: TL_m), or the concentration at which just 50 per cent of the test animals are able to survive, for a specified period of exposure. This exposure period may be 24 hr., 48 hr., or 96 hr. At least the 24-hr. TL_m and the 48-hr. TL_m generally should be determined and recorded, whenever the toxicity is sufficiently pronounced to permit their determination.

b) A median tolerance limit reported may be a concentration at which 50 per cent survival actually was observed in a test (if higher and lower percentages have been recorded for the next lower and higher test concentrations, respectively); or it may be a value derived by interpolation, and based on observed percentages of test animals surviving at concentrations which were lethal to more than half and to less than half of the animals used as test subjects.

c) A simple recommended procedure for derivation of the median value by interpolation involves merely plotting the experimental data on semi-logarithmic coordinate paper, with test concentrations laid off on the logarithmic scale and survival percentages on the arithmetic scale; connecting with a straight line the two points which represent survival percentages at two successive concentrations of the test series which were lethal to more than half and to less than half of the test animals; and noting the concentration which corresponds to 50 per cent survival on this graph. This method may be referred to as straight-line graphical interpolation. It is illustrated in Appendix III. If the difference, or interval, between the two important test concentrations is not much more than 25 per cent of the higher value, an estimate of the median tolerance limit which is accurate enough for most practical purposes can be obtained, even when the observed survival percentages at these concentrations are 0 per cent and 100 per cent. At least one higher concentration and one lower concentration always should be included in the series of concentrations tested, in order to obtain confirmatory data.

d) Other widely accepted methods of estimating the median value more precisely also may be employed when the nature of the available experimental data is such as to clearly justify their use. These include the graphical method which involves fitting a smooth sigmoid curve to the data plotted in the manner described above.

E. Reporting, Interpretation, and Use of Bio-Assay Results

1. Reporting of Experimental Data and Results

a) The kind of fish used as test animals, their source and average size,

the source of the experimental water and its composition (mineral content), or at least its more important characteristics (for example, total alkalinity, hardness, pH, etc.), and the experimental temperature, always should be stated in the report. Other pertinent data, such as the volume and depth of liquid, the number and weight of fish in each test container, and a summary of the results of chemical determinations made in the course of the experiment (for example, observed dissolved oxygen and pH values), also can be instructive and helpful.

b) A complete report should include, in addition to the median tolerance limits determined, a concise statement or table of the experimental data on which these values are based; that is, the numbers of fish used and the observed percentages of survival at the different test concentrations near and within the critical range.

2. Significance and Practical Application of Test Results

The median tolerance limits are only useful indices of the relative toxicity of the substances tested under certain experimental conditions. They obviously do not represent concentrations which may be deemed safe, or harmless, in fish habitats subject to pollution. Therefore, when estimating safe disposal rates or dilution ratios for industrial wastes, on the basis of the toxicity evaluations, one must apply liberal safety factors. Recommendations concerning the magnitude of allowable margins of safety, as well as other related matters pertaining to waste disposal control, are outside the scope of this report, which deals only with recommended bio-assay procedures. Tentative formulas for the estimation of presumably safe concentrations of toxic wastes have been suggested and discussed elsewhere, but they must be further developed and tested.[7]

III. AUXILIARY METHODS

Only those features with respect to which the auxiliary methods differ from the routine procedures previously outlined are considered here. In all other respects the auxiliary and routine tests are identical. When experimental data are reported, any deviation from the basic routine procedure should be clearly indicated, as the test results and their significance may be considerably influenced thereby. None of the auxiliary procedures having been designed for overcoming all difficulties encountered in conducting routine tests; the purpose of each suggested modification of the routine method should be carefully noted before a selection is made. Some of the auxiliary methods can be variously combined, so as to realize the advantages of each.

A. Controlled Artificial Oxygenation of Liquids during Tests

When excessive biochemical or other oxygen demand of a waste is found to interfere with the evaluation of its toxicity by the routine method, one of the methods for controlled artificial oxygenation of test dilutions which have been developed may be employed. These involve oxygenation partly by means of pure oxygen gas, instead of air alone. The solution of oxygen thus can be accelerated without correspond-

[7] For example, the use of the formula—

$$C = \frac{\text{48-hr. } TL_m \times 0.3}{S^2},$$

in which C is the presumably harmless concentration, and $S = \frac{\text{24-hr. } TL_m}{\text{48-hr. } TL_m}$, or of certain modifications thereof under special circumstances—has been tentatively proposed by Hart, Doudoroff, and Greenbank (5).

ingly increasing the rate of escape of any dissolved volatile substances. The ratio of applied oxygen to air depends on the oxygen demand of the liquid tested and must be adjusted, from time to time, to the varying rate of oxygen consumption. In concurrent control tests, the use of oxygen should not be necessary and should be dispensed with, substituting air for the oxygen applied in the experimental aquaria. The resulting reaeration of the water should always be sufficient for maintaining the required dissolved oxygen concentration in the control test containers.

Before one of these methods is employed, the experimental conditions must be adjusted or standardized so that the loss of any dissolved volatile substance from the liquid in the test container would not be much more rapid than it can be in the course of routine tests without artificial oxygenation. The rate of escape of free carbon dioxide gas (initially about 50 to 100 p.p.m. free CO_2) dissolved in clean soft water with total alkalinity not exceeding 40 p.p.m. as $CaCO_3$, may be used as a criterion. The amount of CO_2 lost from this solution in the course of 24 hr., under the experimental conditions selected, should not exceed two-thirds (67 per cent) of the initial free CO_2 content.[8] The carbon dioxide can be dissolved by bubbling the compressed gas (released from a gas disperser) through the water, and its initial concentration and the amount remaining in solution after 24 hr. determined by any suitable method.

Precise control of oxygenation rates by means of simple and inexpensive apparatus has been achieved in the following manner. The test container used is a large (for example, 5-gal.) glass bottle with a fairly wide mouth. A 3-hole rubber stopper is fitted with two ordinary glass tubes (for example, 7 mm. O.D.) extending nearly to the bottom of the bottle. Through these tubes compressed air and oxygen can be introduced, for release in the form of large bubbles, at a constant rate regulated by means of suitable pressure-control devices and valves, the third hole in the stopper serving as a vent.[9] To improve visibility in turbid liquids, each tube may be bent slightly in two places, so as to bring its lower vertical tip near the wall of the bottle. The bottle is filled to a fixed level at the base of the neck, so that the surface of the liquid in contact with the overlying atmosphere has a diameter less than one-half the diameter of the bottle. The liquid may be artificially aerated throughout a test by bubbling compressed air through it at a uniform rate which has been found to be permissible (that is, not excessive), judging by the rate of removal of dissolved free CO_2 in the previously prescribed determination. A suitable aeration rate (that is, the number of bubbles released per minute, which can be readily counted with the aid of a stopwatch) for test containers of any given size and shape must be established before the toxicity tests are undertaken. When necessary, the number of air bubbles released per minute from the air tube can be reduced, and bubbles of oxygen gas, released from

[8] This maximum rate of CO_2 removal can also be expressed as a coefficient, k, of the rate of loss of CO_2, defined by

$$\log \frac{E}{E_0} = -k\,t$$

in which E = free CO_2 concentration at any time, t, in excess of requirements for equilibrium with the atmosphere; $E_0 = E$ when $t = 0$; and t = elapsed time, in hours. The value of k should not exceed 0.02. This maximum value is based on experimental studies of the rate of loss of CO_2 from water of 6-in. depth in aquaria with fish, in the absence of any artificial aeration.

[9] A more complete description of this apparatus and its use and pertinent data which have not yet been published can be obtained from the Environmental Health Center, U. S. Public Health Service, Cincinnati, Ohio. A two-stage pressure-reducing valve and needle valve combination is recommended for the accurate regulation of the flow of oxygen from a cylinder.

the other tube, can be substituted to the extent required for satisfying the oxygen demand of the liquid tested. An adequate dissolved oxygen concentration thus can be maintained without increasing the total number of bubbles released per minute, or seriously altering the rate of escape of any volatile waste components. Even when the use of pure oxygen is not necessary for maintaining an adequate dissolved oxygen concentration, the system described here (with air only introduced) can be useful for controlling the reaeration rate accurately and ensuring its constancy during tests.

An effective method for oxygenating test solutions in open aquarium jars has been described by Hart, Weston, and DeMann (6). An interface of adjustable area between the liquid tested and oxygen gas is maintained inside a submerged inverted funnel with perforations near the base. A small impeller inside the funnel gently agitates the liquid, accelerating the absorption and diffusion of the oxygen. Inasmuch as an adequate description of this apparatus, with detailed diagrams, already has been published, it is not repeated here. Each of the above-mentioned methods has some advantageous features. Sometimes it may be satisfactory simply to maintain an oxygen-enriched atmosphere over the still surface of the liquid tested in a large partially-filled bottle or other suitable test container.

B. Initial Oxygenation of Liquids Tested

The methods and apparatus considered above are for maintaining initially adequate concentrations of dissolved oxygen in the liquids tested. The introduction of oxygen compensates for its gradual consumption during the toxicity tests. However, the dissolved oxygen content must be entirely adequate at the beginning of every test. An industrial effluent can be quite oxygen-free, and it also can have a considerable immediate (chemical) oxygen demand, which must first be satisfied. In order to introduce the required amount of oxygen without recourse to artificial aeration or excessive dilution of the waste to be tested, the extra oxygen may be dissolved in the diluent water before this water is mixed with the waste. This can be done by bubbling compressed oxygen gas (released from a disperser) through the water in a tall container. The oxygen content or the immediate oxygen demand of the waste sample should be determined. The required oxygen content of the diluent to be added, or the required minimum degree of dilution with well-oxygenated (supersaturated) water of known oxygen content, then can be readily estimated. Supersaturation of the diluted waste with oxygen at the beginning of the test proper should be avoided.

High concentrations of wastes which cannot be tested easily by any of the auxiliary methods described here, because of excessive oxygen demand or deficiency, obviously are much greater than the maximum concentrations which can be deemed safe in the receiving waters. When slightly lower concentrations of these oxygen-depleting wastes are not demonstrably toxic, toxicity evidently is lacking or is relatively unimportant. Therefore, only these lower concentrations should be tested, or else plans should be made for treatment of the wastes to be discharged (that is, correction of the oxygen demand or deficiency), before any toxicity tests are undertaken. Toxicity tests following thorough aeration of a waste, or of diluted samples, which removes volatile components, sometimes are desirable. They can be useful in connection with the evaluation of proposed treatment processes, identification of toxic materials, and so forth. However, the toxicity of a treated waste should be clearly distinguished from that of the untreated sample, which may be much more toxic.

C. Renewal of Liquids Tested

Another approved modification of the routine procedure involves renewal of the liquids tested, for the purpose of maintaining more or less uniform concentrations of any volatile and unstable toxic components, as well as an adequate dissolved oxygen content. It is recommended especially when there are reasons for believing that the toxicity of the liquids declines rapidly during the course of a test if they are not renewed. This rapid reduction of toxicity can be a result of extreme volatility of some important dissolved substances. It can also be due to destruction, or removal from solution, of some toxic constituents through their gradual oxidation, combination with metabolic products or mucus of the test animals, accumulation or metabolic destruction within the tissues of the test animals, and so forth. The accumulation of metabolic products sometimes may cause also some increase of the toxicity of the liquids tested.

Constant-flow systems, in which the diluted wastes or test solutions are renewed continually (that is, replaced with fresh mixtures flowing constantly into the experimental containers) have proved very useful in research laboratories. With such apparatus, constancy of the composition and oxygen content of the test liquids can be ensured. For practical reasons, however, the use of constant-flow apparatus cannot be generally recommended for routine application. Such apparatus is somewhat complicated and may require considerable attention. Also, a relatively large amount of waste and of diluent water must be available.

Periodic renewal of the liquid tested, at daily or other convenient intervals, presents less difficulties. This can be accomplished by periodically transferring the test animals quickly, by means of a dip-net, to a new test container with fresh liquid. Renewal of the test medium at intervals of 24 hr. is most often both convenient and desirable, but renewal at shorter or longer intervals, such as 12 hr. or 48 hr., also is permissible. When the oxygen demand of the substance tested is great, artificial oxygenation of the liquids tested, in the manner already described, may be resorted to in addition to their periodic renewal, in order to maintain an adequate dissolved oxygen concentration. This is not often necessary, however, if the liquids are renewed at intervals of 24 hr. or less.

D. Tests of Prolonged Duration

The duration of routine tests has been more or less arbitrarily limited to 96 hr. More prolonged tests sometimes are deemed desirable, inasmuch as even acute toxicity does not always cause death within the 96-hr. test period. When some test animals are found to be still alive, but dying or evidently affected after a 96-hr. exposure to some concentrations of a toxic material, the desirability of prolonging the tests is indicated. Median tolerance limits for longer exposure periods (for example, 8 days, etc.) thus can be determined. The concentration at which half of the animals are killed eventually, while the rest are not seriously affected and survive indefinitely (which has been termed the ultimate median tolerance limit) sometimes can be established. The maximum duration of tests should not exceed, however, the period during which 90 per cent of the controls remain in good condition.

Occasional feeding of test animals during very prolonged tests is recommended. They should be fed sparingly, however. Most fish which previously were well-fed apparently are not seriously affected by fasting for two weeks, or much longer, at the experimental temperatures. Periodic renewal of the liquids tested, which tend to become fouled with excrement and unconsumed food or otherwise gradually altered, also is recommended.

APPENDIX I.—CARE AND FEEDING OF TEST ANIMALS

Keeping fishes in good health in the laboratory is not always easy. Some very delicate fishes, and even some species which are quite hardy in nature, rarely do well in captivity. Patient investigators, however, can learn how to maintain adequate stocks of healthy fishes of various kinds suitable for experimental purposes. Care in the handling of fish during capture and transportation; clean, well-oxygenated, uncontaminated water at a suitable temperature; precautions against infection; and a satisfactory diet are essential requirements.

Seines and other nets used for capturing fish should be of small mesh (usually ¼-in. square, or less). The fish should be handled gently, with wet hands only, and returned to water promptly. Evidently diseased individuals, as well as injured ones which are likely to become diseased, should be promptly discarded, as they may infect healthy fish. Infectious diseases often spread rapidly among fish confined in aquaria.

The water in which fish are held in the laboratory, or transported, must be free of injurious substances, such as chlorine and toxic metals. Free chlorine and chloramine can be removed from treated domestic water supplies by filtration through activated carbon. Contamination of some waters with toxicants can result from sufficiently prolonged contact with metallic surfaces (for example, copper, zinc), certain paints, plastics, and other materials. If fish are to be held in standing water in new concrete or wooden tanks, it may be necessary first to season the tanks thoroughly, changing the water repeatedly before introducing the fish. Glass aquaria are generally most satisfactory for indoor use. Standing water in stock and acclimatizing aquaria, and also in containers used for the transportation of fish, should be continuously aerated with dispersed compressed air, or otherwise. It should be kept clean by frequent removal of unconsumed food and other sediment (by means of a siphon), and complete or partial renewal. Overcrowding of the aquaria should be avoided.

Extreme water temperatures and rapid temperature changes are generally undesirable. When air temperatures are high, cold-water fishes, such as trout and other salmonids, can be transported and kept successfully only in artificially-cooled water, or in running naturally-cool water.

For stocking test animals, several aquaria of moderate size, in which different lots of fish can be kept apart, are preferable to one or two large ones. An epidemic disease may be confined to a single aquarium, and it may be possible to prevent infection of fish held in other aquaria. Badly affected lots of fish generally should be discarded. The aquaria in which they had been kept, as well as the nets used, and so forth, should be disinfected (for example, by means of hypochlorite solution) before introducing new fish. Attempts by inexperienced workers to treat the diseased fish cannot be recommended.

Large stocks of fish should not be maintained in a laboratory for long periods of time if fresh experimental material can be obtained whenever it is needed. Losses due to disease and various mishaps, as well as dietary problems, can be minimized by maintaining only moderate stocks of fish, for use in the near future, whenever this is possible. However, the fresh material obtained from time to time should be kept well apart from healthy fishes that have been in the laboratory for some time. All possible precautions should be taken to avoid infection of the older, acclimated material through careless use of nets, or otherwise, until freedom of the new lot of fish from infectious diseases has been established.

Fishes have varying feeding habits,

many species being chiefly or entirely carnivorous, while some are herbivorous. The natural foods of the test animal species (which can be determined through examination of stomach contents) should be given some consideration when a proper diet is selected. However, substitutes for natural foods, which are satisfactory sustenance for a wide variety of fishes, especially when these are not held in captivity for a very long time, are easily obtainable. The food chosen must be taken readily. It also must be nutritious enough to maintain the fish in apparently good condition considerably longer than the period for which the fish are to be kept in the laboratory prior to their use in toxicity tests. Chronic malnutrition causes anemia, evidenced by paling of the gill color, and gradual emaciation (which can be due also to parasitic diseases, however). Increased susceptibility to infectious diseases and to some toxic substances also can be expected as a result of improper feeding for a long time.

Hatchery-reared fish often can be given artificial food to which they are already accustomed. Most wild fishes can be soon trained to eat artificial foods. These foods may be rejected for some time, but usually are taken when the fish become sufficiently hungry and accustomed to their new environment. Many fishes at first take the artificial food only while it is falling through the water, and not after it has come to rest on the bottom of the aquarium. They should be repeatedly offered a small amount of food at a time, until they learn to eat it readily. Thereafter, the food should be given no more rapidly than it can be mostly consumed before it reaches the bottom. Should the food be continually rejected, some other more attractive and palatable food should be sought.

Most of the fishes which are desirable test animals apparently can be trained to eat and can subsist for a long time on granulated dry foods, which consist of various animal and vegetable matter constituting a balanced animal diet. These convenient foods are easily prepared and stored without refrigeration. Special fish foods can be purchased, but are relatively costly. Certain dry dog foods, such as dog biscuits or pellets, which can be broken up into homogeneous particles of suitable size by crushing or grinding, have proven very satisfactory. The ground or crushed material can be passed through two or more sieves (for example, 20-mesh and 30-mesh) to separate particles of desired size, which can be easily swallowed by the fishes to be fed. The finest material can be discarded or used to make a paste suitable for feeding to some fishes. The material retained on the coarsest sieve can be fed to the largest fish or subjected to further grinding. Dried shrimp, fish, salmon-egg, meat-scrap, and liver meals, as well as other similar meals, also can be fed separately or in various combinations to carnivorous and omnivorous fishes. Cooked and dry cereals are suitable foods for some omnivorous and herbivorous species.

Fresh foods, such as ground fresh fish, shellfish, liver and other internal animal organs, chopped lettuce, and so forth, are desirable additions to the above-mentioned diets. Some fishes may take no other food, or may not thrive when denied fresh foods. Carnivorous fishes sometimes take readily only live foods, such as water fleas (*Daphnia*), other crustaceans, insects, worms, and small minnows. These can be collected from natural waters or cultured. *Daphnia*, an excellent food for small fish, can be very easily cultured in indoor or outdoor tanks, excavations, or small ponds. Usually it is not necessary, however, to provide live foods.

APPENDIX II.—SUGGESTED EXPERIMENTAL CONCENTRATIONS

In order to determine the median tolerance limit, TL_m, any appropriate concentrations of the substance or waste assayed may be tested. It often has been found convenient, however, to select the concentration values from the logarithmic series shown in Table I.

The values in Table I can represent concentrations expressed either as per cent by volume or as parts per million by weight. The values shown may be multiplied or divided, as necessary, by any multiple of 10. For example, the two values in Col. 1 may be 10.0 and 1.0, as shown, or they may be 100 and 10, or 1.0 and 0.1, and so forth. The figures are arranged in five vertical columns in the order (from left to right) in which the corresponding concentrations can be tested for the purpose of gradually delimiting the critical concentration range and locating a median tolerance limit. Each value is approximately midway between the next higher and lower values shown in the preceding columns, when these are plotted on a logarithmic scale.

A series of concentrations, such as 0.1, 1.0, 10 and 100 per cent (Col. 1) may be tested first. Then appropriate concentrations from Col. 2, Col. 3, and so forth, may be tested. A value may be selected from each column which bisects (on a logarithmic scale) the concentration interval, or range, within which a median tolerance limit which is to be evaluated has been shown to be located by preceding tests. For example, if the 24-hr. TL_m and the 48-hr. TL_m of a waste are to be determined, and if it has been found already that all the test animals survive for 48 hr. in a 1 per cent waste dilution, whereas all die within 24 hr. in a 10 per cent dilution, the concentration to be tested next (from Col. 2) is 3.2 per cent. If all the test animals survive for 48 hr. at this concentration, the concentration to be tested next (from Col. 3) is 5.6 per cent.

TABLE I.—A Guide to the Selection of Experimental Concentrations, Based on Progressive Bisection of Intervals on a Logarithmic Scale

Col. 1	Col. 2	Col. 3	Col. 4	Col. 5
10.0	—	—	—	—
—	—	—	—	8.7
—	—	—	7.5	—
—	—	—	—	6.5
—	—	5.6	—	—
—	—	—	—	4.9
—	—	—	4.2	—
—	—	—	—	3.7
—	3.2	—	—	—
—	—	—	—	2.8
—	—	—	2.4	—
—	—	—	—	2.1
—	—	1.8	—	—
—	—	—	—	1.55
—	—	—	1.35	—
—	—	—	—	1.15
1.0	—	—	—	—

Time can be saved and greater uniformity of the tests achieved, however, by performing concurrent tests of several appropriate concentrations shown in two or more successive columns (for example, Cols. 1 and 2, or Cols. 2 and 3). Thus, 3.2, 1.8 and 5.6 per cent dilutions of the hypothetical waste used as an example above may be tested simultaneously with advantage.

The critical concentration range having been adequately delimited by any one of the procedures suggested above, only those concentrations in the right-hand columns of Table I which are apparently within this range need be tested. It is not always necessary to test concentrations shown in Col. 5. However, increased accuracy often can be achieved by testing appropriate concentrations in this column, especially when the critical concentration range has been found to be relatively narrow. For example, if no test animals live for 24 hr. in a 5.6 per cent waste dilution, whereas all survive for 48 hr. in a 3.2 per cent dilution, all intermediate concentrations in Cols. 4 and 5 (namely, 4.2, 3.7,

TABLE II.—A Guide to the Selection of Experimental Concentrations, Based on Decilog Intervals

Concentrations		Log. Conc.
Col. 1	Col. 2	
10.00	—	1.00
—	7.94 (or 7.9)	0.90
6.31 (or 6.3)	—	0.80
—	5.01 (or 5.0)	0.70
3.98 (or 4.0)	—	0.60
—	3.16 (or 3.15)	0.50
2.51 (or 2.5)	—	0.40
—	1.99 (or 2.0)	0.30
1.58 (or 1.6)	—	0.20
—	1.26 (or 1.25)	0.10
1.00	—	0.00

and 4.9 per cent) can well be included in the series of test concentrations.

Some investigators have preferred other similar series of concentrations tested concurrently, such as the series shown in Table II. When feasible, concurrent tests of concentrations shown in both Col. 1 and Col. 2 of Table II may be performed. However, concentrations shown in Col. 1 may be tested first. If additional tests are deemed advisable for reducing the intervals between the test concentrations within the critical range so determined, only the appropriate critical concentrations from Col. 2 then may be tested. The reason for the selection of the concentration values shown in Table II will be apparent when the logarithms of the numbers, tabulated in the right-hand column, are considered. Other series of test concentrations can be equally satisfactory, but a geometric progression of concentration values is always advantageous and should be approximated.

The magnitude of suitable intervals between the concentrations tested for establishing a median tolerance limit by interpolation cannot be definitely prescribed. It depends on the required degree of accuracy (which varies with the planned use of the test results) and on the nature of the experimental data. When two test concentrations, of which one is above and one is below the median tolerance limit, have proved unquestionably lethal to some (for example, 20 per cent or more), but not all of the test animals, determination of survival percentages at intermediate concentrations generally is not essential. Otherwise, the accuracy and reliability of the estimate of TL_m can often be markedly improved by reducing the intervals between the test concentrations. However, the testing of more concentrations may not be justifiable when the increased accuracy thus achieved can have no practical import. The intervals between the concentrations included in Table II (Cols. 1 and 2), and even those included in the first four columns only of Table I, are believed to be sufficiently small under most circumstances. It will be noted that the difference between any one of these concentrations and the next higher concentration of the series is not much more than one-fourth, nor much less than one-fifth, of the higher value. Much narrower and wider intervals are recommended only when they are clearly advantageous and adequate.

APPENDIX III.—EXAMPLE OF THE DERIVATION OF MEDIAN TOLERANCE LIMITS BY STRAIGHT-LINE GRAPHICAL INTERPOLATION

Figure 1 illustrates the estimation of median tolerance limits by the straight-line graphical interpolation method. Hypothetical experimental data are presented in Table III, which shows the numbers of test animals used for testing different dilutions of a liquid industrial waste or effluent, and the numbers surviving after exposure periods of 24 and 48 hr. These experimental results are plotted in Figure 1. The 24-hr. and 48-hr. me-

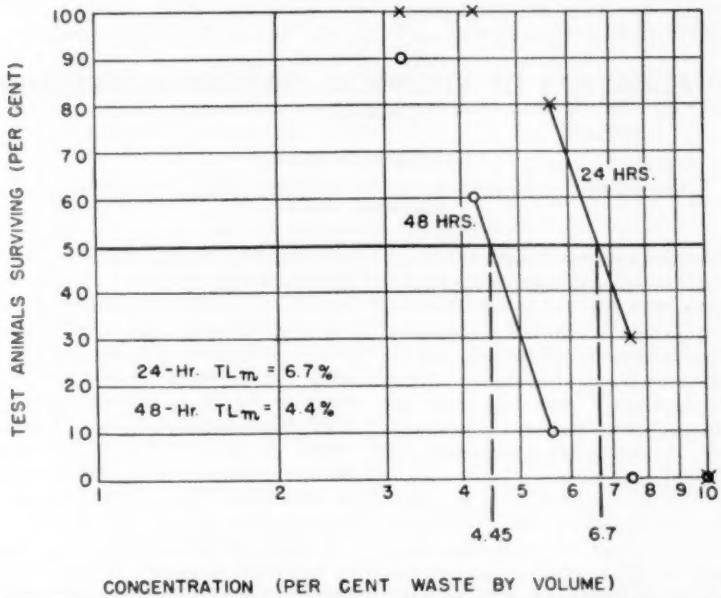

FIGURE 1.—Estimation of median tolerance limits by straight-line graphical interpolation. (Data derived from Table III.)

TABLE III.—Experimental Data (Hypothetical) Plotted in Figure 1

Conc. (% waste by vol.)	Number of Test Animals	Number of Test Animals Surviving After	
		24 Hr.	48 Hr.
10.0	10	0	0
7.5	10	3	0
5.6	10	8	1
4.2	10	10	6
3.2	10	10	9

dian tolerance limits are shown to be about 6.7 and 4.4 per cent waste, respectively.

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EVALUATION OF CHEMICAL OXYGEN CONSUMED TESTS *

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Everyone concerned with the treatment of sewage and industrial wastes is familiar with the B.O.D. (biochemical oxygen demand) test and is aware of its significance, its advantages, and its disadvantages. It is this last term, "disadvantages," that has been the cause of a great amount of investigation in the search for a substitute for the B.O.D. procedure.

By way of emphasis, the principal objection to the B.O.D. test is the length of time involved to reach the final answer, but other objections include the effect of toxic substances, such as heavy metals and bactericidal agents (for example, phenols and cyanides); and also the variable relationship between the 5-day and ultimate oxygen demand, which has its effect on the reliability of the results obtained.

Many substitute tests have been proposed. One of the early ones was the methylene blue relative stability test, which is a very simple method that reduces the over-all time in the case of strong wastes, but the time is increased, rather than decreased, in the case of samples lower in oxygen demand. Also, this test is not considered to be very reliable and is not used to any great extent today, except where no other facilities are available to measure the relative strengths of the waste.

Another test that was introduced early in the picture was the "oxygen consumed from permanganate." This test, which is still the official test as far as "Standard Methods" (1) is concerned, is somewhat involved, but its principal disadvantage arises from its poor coordination with the B.O.D. results. It has been observed that the ratio of B.O.D. to oxygen consumed from permanganate may vary from extremes of about 1:1 to 12:1. However, it should be noted that the ratio for any given sample held to a much more constant value.

Because of the changing rate effect in biological removal of organic material, it was obvious that any method offering a pronounced advantage as to over-all time required would be a chemical method of measurement.

In addition to potassium permanganate as a source of oxygen for the chemical demand, at least three other compounds have been given serious study. Two of these, ceric sulfate and iodic acid, have not proven too satisfactory for one reason or another, but the third, potassium dichromate, has been used very extensively in various modifications, and seems to be the most serious if not the only current contender for a suitable supplement to the B.O.D. test.

It is a personal opinion that it will be quite some time before the B.O.D. test is replaced entirely by some other procedure. It is believed, however, that once a satisfactory supplemental test is adopted, the number of B.O.D. tests can be greatly reduced, probably to the point of making periodic comparisons to assure that the ratio between the particular oxygen consumed tests and the B.O.D. results for a particular waste has not changed.

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Since the conditions under which the chemical oxidation is carried out can be varied over a wide range, the results obtained will also vary widely, and if any one method checks with the B.O.D., it is coincidental; the same conditions would have little chance of agreeing so closely in another type of waste. In order to achieve any semblance to reproducibility in any test, the conditions must be explicit and must be rigorously followed; there must be no deviation in (1) chemical oxidant used, (2) catalyst added, (3) concentration of acid medium, (4) time of contact, (5) conditions of heating, or (6) methods of measurement.

Perhaps the most extensive study of various oxygen consumed methods was carried out by Moore (2) in which he compared the oxygen consumed from permanganate (1); the iodic acid method as proposed by Dzyadzio (3) and by Johnson (4); and various dichromate methods, as outlined by Madison (5), by Rhame (6), and modified by Ingols (7), and also the method proposed by Moore himself in 1948 (8). From the extensive data presented by Moore (9) the best agreement in general was between the Moore dichromate method and the Ingols dichromate method, although the oxygen consumed from permanganate procedure gave less deviation between replicates than any other method than Moore's. The Madison dichromate method and the iodic acid method also gave such a wide deviation between replicates that they were not continued. On the basis of evidence given in these studies, the Ohio Department of Health Laboratories has adopted the Moore (8) dichromate procedure as the standard procedure, and it is understood that many other laboratories are using this method also.

Although the Moore modification is given in the literature (8) this may not be available to all. Because it is believed that quite a few laboratories may be interested in trying the oxygen consumed test for their own information, the procedure given by Moore has been outlined as follows:

Chemical, or Dichromate Oxygen Demand

Reference: Moore, *et al.* Anal. Chem., **21**, 953 (Aug., 1949).

1. Apparatus

a. Reflux condenser, Friedrichs, 24/40 joint.
b. Soxhlet flasks, 300 ml., 24/40 joint.
c. Hot plate, electric.
d. Ring stand, condenser clamp, and ring.
e. Burette and burette support.
f. Dropping bottle.
g. Volumetric flasks, pipettes, and beakers.

2. Reagents

a. 0.2500 *N* potassium dichromate (12.2588 g. per liter).
b. 0.2500 *N* ferrous ammonium sulfate, $Fe(NH_4)_2(SO_4)_2 \cdot 6\ H_2O$. Dissolve 98.0365 g. in 500 ml. of distilled water, add 50 ml. of conc. sulfuric acid, and dilute to 1,000 ml. Standardize against dichromate solution (see "Discussion").
c. Ortho-phenanthroline indicator. Dissolve 1.485 g. of ortho-phenanthroline and 0.695 g. of ferrous sulfate ($FeSO_4 \cdot 7H_2O$) in 100 ml. of distilled water.
d. Conc. sulfuric acid, C.P.
e. Standard silver nitrate solution. Dissolve 2.400 g. of silver nitrate in 1,000 ml. of distilled water. Standardize against sodium chloride. Adjust the silver solution so that 1 ml. = 1 mg. chloride.
f. Potassium chromate indicator. 5 per cent solution.

3. Procedure

a. Dilute the appropriate volume of sample with distilled water to 50 ml.

b. Prepare a blank using 50 ml. of distilled water.

c. To the sample and blank, add 25 ml. of the N/4 dichromate solution and 75 ml. of conc. H_2SO_4. (Note: Use care in adding acid and mix thoroughly.)

d. Reflux each preparation for 2 hr. Cool and transfer to beakers or Erlenmeyer flasks. Rinse Soxhlet flasks thoroughly and dilute each sample to about 300 ml.

e. Add 1 or 2 drops of the o-phenanthroline indicator and using N/4 ferrous ammonium sulfate solution as the titer, titrate to the proper end point (see "Discussion").

4. Calculations

a. B = Volume of N/4 $Fe(NH_4)_2(SO_4)_2$ for blank;
S = Volume of N/4 $Fe(NH_4)_2(SO_4)_2$ for sample;
N = Normality of $Fe(NH_4)_2(SO_4)_2$;
A = Volume of sample used in 3a, then

$$\text{p.p.m. Oxygen Consumed} = \frac{8{,}000\ N\ (B\text{-}S)}{A}.$$

b. If chlorides are present, a determination should be made, and the oxygen consumed value corrected, as follows:
p.p.m. Oxygen Consumed = Oxygen consumed, apparent − (0.215 × p.p.m. chlorides).

5. Discussion

In standardizing the N/4 ferrous ammonium sulfate against the N/4 dichromate solution, take a 10- to 25-ml. portion of the dichromate and add about 5 ml. of conc. H_2SO_4, as in a sample. Unless acid is added, no indicator color change will take place.

The o-phenanthroline indicator solution is a deep bright red. When the titration is about one-third to one-half complete, a change to green will be noted. The correct end point is an abrupt change from this green to red.

The method above uses sufficient sulfuric acid to oxidize the chlorides, if present, and the correction must be made as directed. If less acid is used, the chlorides may or may not be oxidized, introducing an error that cannot be corrected by calculation.

(Note: The o-phenanthroline may be obtained from G. Fredrich Smith Chemical Co., 867 McKinley Ave., Columbus 12, Ohio. The catalog number is No. 163 and the price is $2.25 per gram, or $10.00 for 5 grams.)

The procedure involves the refluxing in suitable apparatus of a mixture of the waste, using an aliquot portion, diluted to 50 ml. with distilled water to which have been added 25 ml. of N/4 potassium dichromate and 75 ml. of concentrated sulfuric acid. After 2 hr. reflux time, the mixture is cooled, diluted to approximately 300 ml., and the excess dichromate is determined by titration with N/4 ferrous ammonium sulfate, using a orthophenanthroline-ferrous sulfate complex (ferroin) as an internal indicator. Moore did not state the amount of concentrated sulfuric acid added to stabilize the standard ferrous ammonium sulfate, so the Ohio Department of Health started to use 5 per cent by volume, whereas Moore since has recommended 2 per cent. The lower acid concentration has the disadvantage of allowing the standard to decrease in normality more rapidly, but in all cases the strength should be checked sufficiently often to assure accuracy. The purpose of diluting the mixture before titration is to reduce the sulfuric acid concentration to below 30 per cent to ensure accuracy of the end point. A blank should be run daily, and used in the calculation.

An appreciable source of error arises in the presence of chlorides, which are oxidized in acid medium by the dichromate. Unless the chloride ion is known to be negligible in comparison to the oxygen consumed value, the exact quantity should be determined by the usual titration with silver nitrate in the pres-

ence of potassium chromate indicator, and the correction applied as noted in the procedure.

Various other modifications of this method have been proposed by Moore, who suggested that they be investigated before adoption. These are (a) the use of silver sulfate as a catalyst, (b) the reduction of refluxing time to ½ hr. or 1 hr., (c) the use of larger quantities of standard dichromate, and (d) the use of other than a 50:50 ratio of concentrated sulfuric acid in the refluxing mixture.

In using the oxygen consumed procedure as described, it has been found that for straight sewage wastes the 5-day B.O.D. is, in general, reasonably close to 65 to 70 per cent of the oxygen consumed results. But in industrial waste studies there seems to be no general pattern of relationship for the different wastes. This is due to the greatly variable type of oxidizable compounds present. However, the ratio of oxygen consumed to B.O.D. as found in a series of samples from the same source generally follow the same pattern.

The oxygen consumed test is being used more and more; bearing in mind the time-saving feature, and not forgetting its limitations, it is recommended that it be tried out wherever the necessary equipment is available or can be procured. After correlating the results with the B.O.D. value, the individual operator or chemist can decide on its application to his needs.

TABLE I.—Comparison of B.O.D. and Oxygen Consumed Values for Various Industrial Wastes

Source or Type of Waste	B.O.D. (p.p.m.)	O_2 Consumed (p.p.m.)	Ratio, B.O.D./O_2 Consumed (%)
Sugar beet refinery:			
Process water	5,840	10,980	53
Process water	3,990	9,680	41
Process water	5,270	12,220	43
Process water	5,070	10,680	48
Process water	6,250	11,190	52
Process water	5,103	10,240	50
Flume water	945	1,700	56
Flume water	1,440	2,060	70
Flume water	1,310	1,740	74
Flume water	1,950	2,180	90
Flume water	915	5,440	17
Lime slurry	3,090	16,120	25
Lime slurry	4,490	24,300	18
Flume water sludge	1,930	5,290	37
Flume water sludge	1,090	1,820	60
Flume water sludge	1,110	1,690	66
Flume water sludge	900	1,630	55
Battery supply	750	1,960	38
Discharge to lagoon	2,120	2,635	80
Phenolic wastes:			
Still wastes	1,480	13,820	11
Still wastes	3,900	6,500	60
Quench return	1,610	3,360	48
Quench return	320	8,290	3
Plant sewer	585	1,100	53
Plant sewer	1	905	<1
Dephenolizer inlet	7,800	13,150	60
Dephenolizer outlet	4,730	12,740	37
Miscellaneous	3,150	14,400	22
Miscellaneous	1,350	2,990	45
Miscellaneous	810	1,850	44
Food processing:			
Canneries	837	1,270	66
Canneries	2,090	16,020	13
Canneries	8,180	12,820	64
Canneries	880	2,200	40
Canneries	504	1,060	48
Slaughterhouse	815	4,030	20
Slaughterhouse	630	770	82
Miscellaneous	2,270	3,490	65
Miscellaneous	313,000	540,000	58
Miscellaneous	520,000	613,000	85
Miscellaneous	248,000	327,000	76
Oil refinery	236	464	51
Oil refinery	77	164	47
Oil refinery	92	156	59

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COLORIMETRIC DETERMINATION OF CYANIDE

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In recent years attention has become focused on the need of determining cyanide in sewage and electroplating wastes, one of the major problems in sewage analysis today being the determination of trace amounts of this constituent. This emphasis on the determination and control of free cyanide in the range of 0.1 to 20 p.p.m. has arisen with the realization of the toxicity of cyanide to fish and microorganisms. Standards for effluents and polluted waters are now coming into use; these standards make methods of determining cyanide in this concentration range a necessity.

The problem of determination and control is complicated by complex cyanides, which, if not actually toxic themselves, are potential sources of free cyanide. For the latter reason, this investigation was divided into the determination of total and of free cyanide.

Total cyanide consists of all inorganic cyanides, present either as hydrogen cyanide, in ionic form, or in complexes such as ferricyanide. Free cyanide is taken to mean cyanide as found in hydrogen cyanide, alkali cyanides, or metal cyanides, such as nickel cyanide, which are in equilibrium with hydrogen cyanide.

For the determination of total cyanide the preliminary destruction of the cyanide-containing complexes is necessary. Once the cyanide is present in the free state, it can be titrated with silver nitrate according to the method of Liebig, or determined colorimetrically. Since the end point of the titration is observed by the formation of a precipitate, no other precipitates should be present. This necessitates the separation of the cyanide from the sample. For the colorimetric system separation is also necessary. A method was developed, therefore, whereby the cyanide was distilled from mixed phosphoric and citric acids. This treatment serves to destroy any complexes and to separate the cyanide from thiocyanate and cations, which interfere with the colorimetric determination. For cyanide concentrations greater than 50 p.p.m., titrimetric methods are applicable; for smaller concentrations a suitable aliquot is taken and the cyanide determined colorimetrically.

Although several colorimetric methods having the required sensitivity of 0.1 to 20 p.p.m. are listed in the literature (1) (2) (3) (4), none is directly applicable for the determination of cyanide. Of the methods listed, the use of the benzidine-pyridine (1) or pyridine-pyrazolone (2) reagent with cyanogen halide appeared the most promising, as these two methods require a minimum of preliminary treatment of the sample and also do not require concentration or volatilization. Of these two, the pyridine-pyrazolone reagent was chosen for color development because of its sensitivity and because the color system was found to be more stable and sensitive than that developed with a benzidine-pyridine reagent.

Determination of Total Cyanide

Cyanide is separated from metals, thiocyanate, and most of the organic matter in sewage by distillation from a phosphoric acid solution. In this

process complex cyanides are decomposed. The isolated cyanide is then determined colorimetrically using chloramine T and a pyridine-pyrazolone reagent.

1. Apparatus

1.1. Photoelectric filter photometer transmitting at 620 mμ., or photoelectric spectrophotometer.

1.2. Distilling flask, 1,000-ml.

1.3. Condenser, Allihn or straight, 200-mm.

1.4. Adapter.

1.5. Separatory funnel, 125-ml.

The distillation apparatus is assembled by fitting the separatory funnel in the distilling flask so that the sample may be added during the distillation. The condenser, with the adapter, is attached to the sidearm of the distilling flask. A piece of 6- or 7-mm. glass tubing, which reaches below the surface of the receiving solution, is fitted to the adapter with Tygon tubing.

2. Reagents

2.1. Alkaline solution of ethylenediamine tetracetic acid, 10 per cent. Dissolve 10 g. c.p. "versene acid" (Bersworth) and 5.6 g. sodium hydroxide in 85 ml. of water.

2.2. Chloramine T, 1 per cent. Dissolve 0.20 g. of chloramine T (Eastman) in 20 ml. of water. The solution should be freshly prepared each day.

2.3. Citric acid, c.p.

2.4. Phosphoric acid, 85 per cent, c.p.

2.5. 1-Phenyl-3-methyl-5-pyrazolone (Eastman). The pyrazolone is recrystallized twice from 95 per cent ethanol, and can be stored for months in a dark bottle.

2.6. Bis-(1-phenyl-3-methyl-5-pyrazolone). Dissolve 17.4 g. of the twice recrystallized (from 95 per cent ethanol) 1-phenyl-3-methyl-5-pyrazolone in 95 per cent ethanol. Add 25 g. of freshly distilled (under reduced pressure) phenylhydrazine and reflux the mixture for at least 4 hr. The insoluble portion, which is filtered off and washed with hot ethanol, is bis-(1-phenyl-3-methyl-5-pyrazolone). It is stable and can be stored for long periods in a dark bottle.

2.7. Pyridine-pyrazolone reagent. Twice recrystallized 1-phenyl-3-methyl-5-pyrazolone is added to 125 ml. of hot water. An excess of the pyrazolone should be present so that a saturated solution is formed. The solution is stirred and allowed to cool to room temperature, and the excess pyrazolone filtered off. To this solution add 25 ml. of redistilled pyridine containing 0.025 g. of the bis-pyrazolone. The bis-pyrazolone can be dissolved in the pyridine by stirring. The pyridine and aqueous pyrazolone solutions should be freshly prepared and mixed only shortly before use.

2.8. Sodium acetate, 10 per cent. Dissolve 10 g. of $Na_2C_2H_3O_2 \cdot 3H_2O$ in 90 ml. of water.

2.9. Standard cyanide solution. Dissolve 2 g. of c.p. sodium cyanide in a liter of solution. This solution is then standardized by titrating with 0.05 N standard silver nitrate according to the Liebig method, using ammonium hydroxide and potassium iodide to indicate the end point. The standard solution is prepared from this solution by 100-fold dilution.

2.10. Water, cyanide- and metal-free, for the preparation of reagents and dilutions. This water can be prepared by distillation from alkaline permanganate or by passing water through a de-ionizer.

3. Procedure

Preparation of Standard Curve

3.0. Dilute 20-, 10-, 5-, 3-, and 1-ml. portions of the standard solution to 250 ml., add 10 ml. of versene solution, and distill the cyanide in the manner described below. Use a 25-ml. aliquot

of the distillate obtained (3.4.) for color development. For a good approximation, however, a standard curve can be prepared by diluting the standard solution 10-fold, taking 10-, 5-, 3-, 2-, 1-ml. aliquots and developing the color as described below (3.5.). Plot milligrams of cyanide *vs.* absorbancy.

Analysis of Sample

3.1. Filter the sample to remove silt and insoluble material, and wash the precipitate thoroughly. Add 10 to 15 ml. of the versene solution (2.1.) to 250 ml. of the sample containing 0.03 to 10 mg. cyanide, either as the ion or complexed with metal ions. A precipitate may form on the addition of the versene, but this does not interfere in the determination.

3.2. Add about 0.3 g. of citric acid (2.3.) and 35 ml. of the phosphoric acid (2.4.) to approximately 100 ml. of water in the distilling flask and close the stopcock in the separatory funnel. Add enough water to the receiving flask to cover the end of the tube from the adapter into the receiving flask by at least 0.5 cm. Add 10 ml. of the sodium acetate solution (2.8.) to the receiving solution. With the cooling water to the condenser shut off, heat the solution in the distilling flask to boiling until the condenser becomes warm. Next allow the water in the condenser to circulate and adjust the height of the column of water in the delivery tube to 20 to 35 cm. with the stopcock in the separatory funnel. The solution in the distilling flask is kept boiling.

3.3. Slowly add the sample from the separatory funnel to the boiling solution. The boiling should be continued and the reduced pressure maintained. As soon as the sample has been added, close the stopcock of the separatory funnel. Rinse the sample container, as well as the separatory funnel, with about 75 ml. of distilled water and add these washings to the distilling flask. After all the washing solution has been added, boil the solution in the distilling flask until only about 40 ml. remain. At this point the phosphoric acid is practically concentrated. Turn off the heat and admit air through the separatory funnel by opening the stopcock. The end point of the distillation is quite easily detected by the fact that the color of the solution begins to change to brown and the viscosity increases rapidly.

3.4. Drain any remaining liquid from the condenser into the receiving flask. If organic materials are present in the distillate, they can be removed by extraction with benzene or carbon tetrachloride. Transfer the distillate, which must be within pH 5 to 8 (adjust with dilute acetic acid or sodium acetate), to a 500-ml. volumetric flask. Wash out the receiving flask, and dilute the distillate and washings to 500 ml.

3.5. Transfer an aliquot of 1 to 25 ml., containing 0.001 to 0.010 mg. of cyanide, to a 50-ml. volumetric flask. Add 0.3 ml. of chloramine T solution, and allow it to stand for one minute. Add 15 ml. of the pyridine-pyrazolone reagent (2.7.) and let the solution stand for 30 min. Then adjust the volume to 50 ml. and measure the absorbancy of the blue color. From the standard curve, obtain the amount of cyanide in the aliquot used, and from this calculate the cyanide concentration in the original sample.

$$\text{p.p.m. CN} = \text{mg. cyanide} \times \frac{500}{\text{aliquot}} \times \frac{1{,}000}{\text{ml. sample}}$$

Determination of Free Cyanide

Free cyanide is separated from metals, thiocyanate, and complex cyanides by extraction as hydrogen cyanide from acid solution with isopropyl ether.

Thiocyanate concentrations below 25 p.p.m. show no interference. At higher concentrations they also react with the reagent to form the desired color, but

only to a minor degree, so that 200 p.p.m. thiocyanate showed an absorbancy equivalent to 1 p.p.m. cyanide. The hydrogen cyanide is re-extracted from the ether with sodium hydroxide, the hydroxide neutralized with acetic acid, and the color developed in the neutralized solution using chloramine T and the pyridine-pyrazolone reagent. If organic materials are found to interfere in the color development, they are removed by extraction with benzene, petroleum ether, or carbon tetrachloride prior to the extraction of the free cyanide with ether.

1. Apparatus

1.1. Photoelectric filter photometer transmitting at 620 mμ., or photoelectric spectrophotometer.

1.2. Separatory funnels, 125-ml. and 250-ml.

2. Reagents

2.1. Acetic acid, 5 per cent. Mix 10.0 ml. of glacial acetic acid with 190 ml. of water.

2.2. Benzene.

2.3. Chloramine T, 1 per cent. Dissolve 0.20 g. of chloramine T (Eastman) in 20 ml. of water. The solution should be freshly prepared each day.

2.4. Diisopropyl ether. Purify the ether by extracting with 5 per cent sodium hydroxide solution. Wash the ether with two portions of water. In each of the extractions allow at least 5 min. for the layers to separate. The isopropyl ether used in the determination can be repurified in this manner.

2.5. 1-Phenyl-3-methyl-5-pyrazolone (Eastman). The pyrazolone is recrystallized twice from 95 per cent ethanol, and can then be stored for months in a dark bottle.

2.6. Bis-(1-phenyl-3-methyl-5-pyrazolone). Dissolve 17.4 g. of the twice recrystallized 1-phenyl-3-methyl-5-pyrazolone (2.5.) in 100 ml. of 95 per cent ethanol. Add 25 g. of freshly distilled phenylhydrazine and reflux the mixture for at least 4 hr. The insoluble portion, which is filtered off and washed with hot ethanol, is bis-(1-phenyl-3-methyl-5-pyrazolone). It is stable and can be stored for long periods in a dark bottle.

2.7. Pyridine-pyrazolone reagent. Twice recrystallized 1-phenyl-3-methyl-5-pyrazolone is added to 125 ml. of hot water. An excess of the pyrazolone should be present so that a saturated solution is formed. The solution is stirred and allowed to cool to room temperature, and the excess pyrazolone is filtered off. To this solution add 25 ml. of redistilled pyridine containing 0.025 g. of the bis-pyrazolone. The bis-pyrazolone can be dissolved in the pyridine by stirring. The pyridine and aqueous pyrazolone solution should be freshly prepared and mixed only shortly before use.

2.8. Sodium hydroxide, 3 per cent. Dissolve 30.0 g. of sodium hydroxide in 970 ml. of water.

2.9. Standard cyanide solution. Dissolve 2 g. of c.p. sodium cyanide in a liter of solution. This is then standardized by titration with 0.05 N standard silver nitrate according to the Liebig method, using ammonium hydroxide and potassium iodide to indicate the end point. The standard solution is prepared from this solution by 100-fold dilution.

2.10. Water. Cyanide-free distilled water is needed for the preparation of reagents and dilutions. The water can be prepared by distillation from alkaline solution or by passing water through a de-ionizer.

3. Procedure

Preparation of Standard Curve

3.0. Dilute 20-, 10-, 5-, 3-, 2-, and 1-ml. portions of the standard solution to 50 ml. and treat by the procedure described below. A separate calibration curve is needed for the method using the extraction with benzene prior to the extraction with ether.

Analysis of Sample

3.1. Filter the sample; or allow solids to settle out. Measure out a 50-ml. aliquot into a 125-ml. separatory funnel.

3.2. If the sample contains organic matter, which will interfere in the color formation with the pyrazolone reagent, add 40 ml. of benzene to the sample. Isooctane, petroleum ether, or carbon tetrachloride may be used in place of benzene. If the sample solution is basic, neutralize it to phenolphthalein with 5 per cent acetic acid. Shake the two layers and allow them to separate. Transfer the aqueous phase to another 125-ml. separatory funnel containing 40 ml. of the isopropyl ether. Wash the benzene with two 8-ml. portions of water, and add these washings to the rest of the sample. Discard the benzene.

3.3. Acidify the sample with 2 ml. of glacial acetic acid. Shake the two layers, allow them to separate, drain the aqueous phase into another 125-ml. separatory funnel, and transfer the ether layer to a 250-ml. separatory funnel also. Repeat the process with three more 40-ml. portions of isopropyl ether, collecting all the ether in the 250-ml. separatory funnel. Discard the aqueous phase.

Drain any water from the ether layer, and then add 8 ml. of 5 per cent acetic acid. Shake the mixture, allow the acid to settle out, and discard it. Repeat this process with another 8 ml. of 5 per cent acetic acid. Let the ether layer stand for 5 min. and then drain off any acid that separates out.

Extract the hydrogen cyanide from the ether layer with three 32-ml. portions of 3 per cent sodium hydroxide. Collect these sodium hydroxide extracts in a 100-ml. volumetric flask and dilute to volume with 3 per cent sodium hydroxide.

3.4. Transfer a 10-ml. aliquot to a 50-ml. volumetric flask. Add 9.0 ml. of 5 per cent acetic acid to this aliquot to adjust the pH to 5.5 to 8, and then add 0.3 ml. of chloramine T solution (2.3.), stopper and shake the resulting solution, and allow it to stand for 1 min. In place of the 10-ml. aliquot a 5- or 25-ml. aliquot of the hydroxide solution may be used. In the former case 4.5 ml. of 5.0 per cent acetic acid, and in the latter case 7.5 ml. of 15.0 per cent acetic acid, should be used to adjust the pH. The treatment with chloramine T remains the same. After the 1-min. period has elapsed, quickly add 15 ml. of the pyridine-pyrazolone reagent (2.7.), and adjust the volume to 50 ml. Measure the absorbancy of the sample after a 30-min. period to allow the blue color to develop. Water can be used as the blank. From the standard curve obtain the amount of cyanide in the aliquot used, and from this calculate the cyanide concentration in the original sample.

$$\text{p.p.m. CN} = \text{mg. CN} \times \frac{2{,}000}{\text{aliquot}}$$

3.5. Note: In case no materials which will interfere in the color development are present, the extraction with benzene (3.2.) may be omitted, and the sample treated directly as in 3.3.

Acknowledgment

The authors wish to express their appreciation to the United States Public Health Service for financial support of this project through a research grant from the National Institutes of Health, and to the Federation of Sewage and Industrial Wastes Associations, under whose auspices the research was conducted. Special thanks are due W. D. Hatfield, the coordinator for the project.

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POLLUTION PATTERNS IN THE OHIO RIVER

Freshets caused by heavy rains in the Ohio River valley accentuate health hazards from sewage pollution. Bacteria from these human wastes are carried downstream to the water supplies of other cities at high flows faster than the bacteria die off in the stream. This is one of the findings pointed up in a report on "Pollution Patterns in the Ohio River," recently released by the Engineering Committee of the Ohio River Valley Water Sanitation Commission.

This report forms part of the Commission's program to establish baselines from which pollution abatement progress can be measured. In addition, the most polluted stretches of the Ohio River are being charted. Intensified action will be focused on these highly polluted stretches.

The report includes hydrometric, chemical, and mineral studies, as well as the bacteriological findings.

At the same time the Engineering Committee also considered establishment of a permissible limit for phenols in the rivers. Phenols are a primary source of tastes and odors in public water supplies. The Committee is requesting comments from representatives of industry on the technical data assembled in support of this proposed limit for phenols.

"Adoption of a permissible limit for phenols," according to the committee, "will show industry what part it will have to play in protecting public water supplies from phenol tastes. At the same time industry is assured that all phases are evaluated in defining a reasonable use for the river."

Reasonable use of a river, as defined by the Commission, means providing clean and palatable water for municipalities and industries, aquatic life, and recreation, as well as utilizing the dilution capacity of a stream to assimilate wastes that will not interfere with primary stream use.

SOME INDUSTRIAL WASTE TREATMENT PLANTS IN CONNECTICUT *

By Willis J. Snow

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This paper is a general review of several of the more successful industrial waste treatment plants that have been in operation in Connecticut for a number of years.

In such a highly industrialized state as Connecticut the waste disposal problems are many and varied and it has been learned that every factory requires a separate and individual study to determine the most satisfactory solution of its problem. Although it is true that an over-all type of treatment may be applicable to a number of industries carrying on the same general kind of manufacturing processes, it is also true that the inherent characteristics of each mill thwart any effort to prescribe a uniform remedy for each source of pollution. It is these differences and variations which help to complicate the industrial waste disposal program.

The following treats briefly with the treatment of four different but common kinds of trade wastes; namely, pickle liquor from a steel wire mill, plating-room wastes with a predominance of chromium, metallurgical wastes containing cyanide, and white water from paperboard mills.

Pickle Liquor from Steel Wire Mill

The Gilbert and Bennett Manufacturing Company, located at Georgetown, Conn., on a fine trout stream, was founded in 1818 by Benjamin Gilbert, who made his first screen or sieve for sifting meal from bran by weaving together the hairs from a horse's tail, and who later invented and developed wire screens. In 1930, this concern voluntarily installed a waste treatment plant, which has functioned satisfactorily since that time.

Originally, the strong acid pickle liquor, when it became unfit for further use, was run into wooden tanks in a special building where evaporation and crystallization took place, thus producing a good grade of copperas (ferrous sulfate), which had a ready sale. The mother liquor was then returned to the pickling room for re-use.

As time passed, the revenue from the sale of copperas gradually decreased, whereas the cost of carrying on this operation increased until finally, because it was neither profitable nor convenient, the crystallizing process was discontinued and the strong acid liquor was simply neutralized and pumped directly to the sludge lagoons, as described hereinafter.

It is the rinse water from the pickling process which must be treated regularly. To this, in recent years, has been added the acid waste from two galvanizing departments.

All of the acid liquor is run into a circular concrete tank (Figures 1 and 2), where it becomes well-mixed and equalized, thus insuring a more uniform chemical treatment. The tank has a detention period of from 2 to 6 hr., depending upon operations within the mill. From here it passes into a small sump or wet well, at a fairly constant rate, where it is treated with a lime slurry made by adding hydrated lime to river water in a chemical mixing

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tank. The lime dosage is controlled by means of an adjustable ratchet, which regulates the speed at which the feed screw revolves, while the volume of the river water is controlled by means of a globe valve. Because of uniformity of conditions in the mill and efficient equalizing of the acid liquors it is seldom necessary to change the adjustment of the dosing mechanism. The equalized wastes are tested at least four times a day to insure adequate treatment.

From the wet well a plunger pump transfers the treated liquor to a 5,288-gal. mixing tank located about 400 yd. downstream from the receiving unit. The agitator is fitted with paddle-type stirrers, which revolve at a moderate rate of speed (about 120 r.p.m.) Additional lime can be added at this point in case of an emergency.

After complete mixing in the agitator, the liquor, which now contains a distinct floc, is run into a clarifier equipped with a sludge-removing mechanism. The capacity of the clarifier is 60,000 gal.; the flowing-through period is seven or more hours, depending upon production in the mill. The effluent from the clarifier is discharged directly into the Norwalk River.

Sludge Lagoons

The settled sludge is discharged into two lagoons having a total area of approximately three acres. The lagoons have no underdrains, but the bottom consists of a layer of cinders and each is surrounded by a cinder dike built on a stone foundation. The dikes are at least 12 ft. thick and are very carefully maintained. The wet sludge is pumped into the first lagoon, from whence it overflows to the second lagoon. These units have an average depth of about 6 ft.

It is possible to by-pass the clarifier and turn the treated wastes directly into the lagoons. The plant can operate in this manner for several weeks without encountering any difficulty. However, this procedure is followed only when it becomes necessary to repair the clarifier mechanism or when treating the strong pickle liquor previously mentioned.

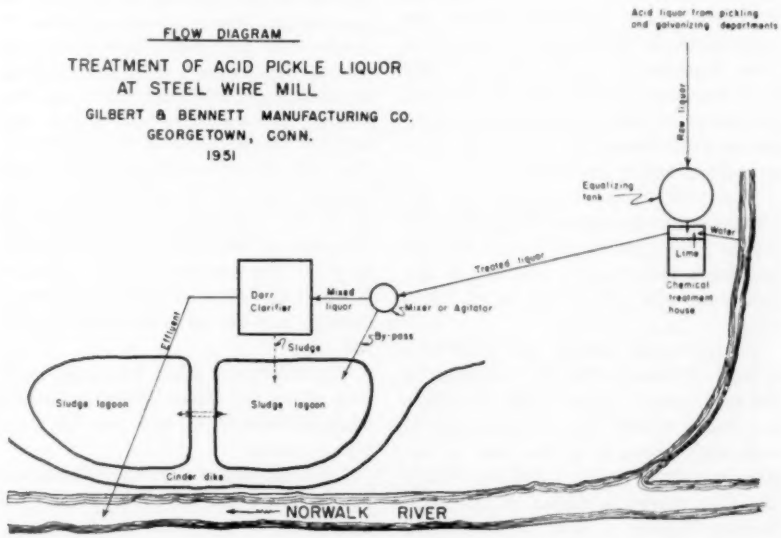

FIGURE 1.

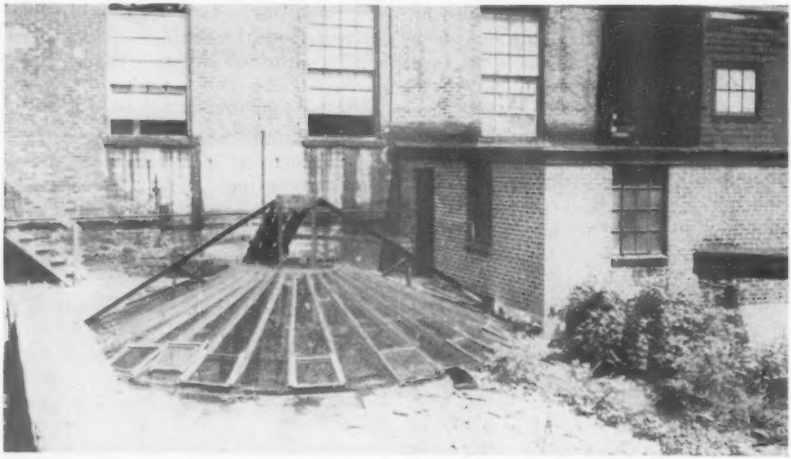

FIGURE 2.—Equalizing tank and chemical treatment building, Gilbert and Bennett Manufacturing Co., Georgetown, Conn.

Although the water which drains out of the sludge must seep through the cinder dikes, there is no discoloration on the outside of the dikes or on the river bottom, thus indicating that the treatment is quite effective. The treatment plant was in operation for about 10 years before any of the sludge was removed from the lagoons. Since that time, however, a considerable quantity of it has been carted away to be used for removing sulfur from illuminating gas and the demand for it for this purpose has steadily increased.

The waste liquors handled by the plant amount to approximately 200,000 g.p.d. This liquor is not highly colored, but has a rather dirty and murky appearance. Its pH ranges from 3.0 to 4.2.

As previously stated, hydrated lime is used exclusively for the treatment of the acid liquor. About 3,000 lb. of lime are added to the flow of waste liquors each day, which is at the rate of approximately 15 lb. per 1,000 gal. When the lime slurry is added to the iron-bearing liquors a dark green floc or precipitate commences to form. This floc is clearly discernible in the mixer or agitator.

Results and Cost

As the treated liquor passes through the clarifier the precipitate settles to the bottom to form the sludge, and the supernatant liquor passes over the effluent weir and flows directly to the river. The effluent is clear, colorless, sparkling, and free from solids; its iron content is rarely over 0.2 p.p.m. and the pH varies between 8.2 and 11.2. The pH of the receiving stream runs from 7.2 to 7.6. The pH of the stream below the treatment plant is essentially the same as that above, due to the efficiency of the treatment and adequate dilution, the latter being at least 10 to 1 during periods of minimum stream flow.

The treatment plant has easily handled all of the waste liquors produced when 150,000 lb. of wire per day have been cleaned.

The total cost of operation, including power, labor, and chemicals, is approximately $5,000 per year.

The entire plant is under constant

supervision and chemical control. The effluent is tested at least twice each day with dilute aqua ammonia, and pH determinations are made at the same time. Occasionally, more complete and extensive tests are made on the liquors and sludge.

No serious difficulties have been encountered in the operation of this plant and its efficiency is indicated by the fact that the condition of the Norwalk River immediately below the company property is as good as that above. Fish thrive in the stream apparently unaffected by the liquors discharged into it. In fact, because of its satisfactory condition, the State Board of Fisheries and Game now stocks this river with trout.

There is little doubt that the successful operation of this industrial waste treatment plant has been due to two factors; namely, careful and conscientious attention on the part of the company chemist and interest and cooperation on the part of the company officials. The treatment plant is considered an integral part of the factory and as such is operated with the same care and attention given productive equipment. It is gratifying to the state agencies that all this has been done thoroughly and voluntarily by the officers and personnel of the company, uninfluenced by orders, threats, or coercion.

Plating Room Wastes

The Electrolux Corporation, at Old Greenwich, Conn., does a considerable amount of plating, including chromium, nickel, and cadmium. Chromium plating is the major process. It was the typical yellow or amber rinse water from this process which created a serious pollution problem in the receiving water, especially since this brook flows through a school playground.

In 1940 a treatment plant was constructed consisting of two 20,000-gal. cylindrical wooden settling tanks, chemical mixing and dosing tanks, a filter press, a combination chemical storehouse and laboratory, and other necessary appurtenances. Since then six new settling tanks have been added,

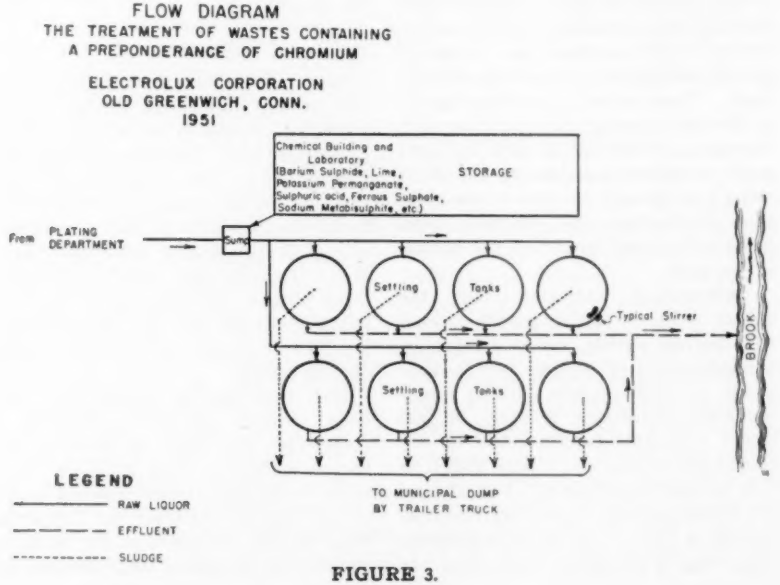

FIGURE 3.

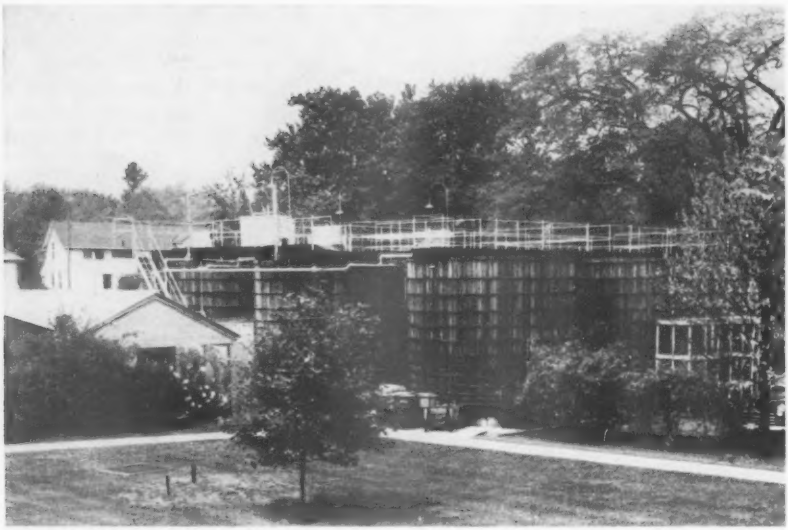

FIGURE 4.—Chromium waste treatment plant at Electrolux Corp., Old Greenwich, Conn., showing settling tanks and (left foreground) chemical building and laboratory.

making a total of eight sedimentation units at the present time (Figures 3 and 4).

The first tanks were equipped with stirring devices consisting of a vertical central shaft to which were attached paddles set parallel to the bottom of the tank. These caused a swirling motion of the liquor rather than a mixing and, therefore, all new tanks were equipped with propeller-type stirrers, the shaft being run through the side of the tank, near the bottom, the propeller itself being set at right angles to the bottom of the tank.

Experiments indicated that the wastes could be satisfactorily treated with barium sulfide, sulfur dioxide, or a combination of sodium sulfide, copperas, and sulfuric acid. Under actual operating conditions, however, it was found that best results could be obtained by treating the wastes with 5 to 6 lb. of barium sulfide and 4 lb. of lime per 1,000 gal., after previous acidification to a pH of 2.0 if the untreated liquor had a pH above that value. It was possible to obtain complete removal of chromium with this method and a lesser removal of the other metals and the cyanide.

Treatment Difficulties

Some difficulties were encountered. The barium sulfide was difficult to dissolve; an increase in the concentrations of copper, iron, and nickel caused the appearance of a black colloidal precipitate in the top water in the settling tanks; and not infrequently a disagreeable hydrogen sulfide odor was liberated. Occasionally, additional treatment with a reducing agent, such as potassium permanganate, was required. In 1944 it became difficult to obtain barium sulfide.

For these various reasons the company started using ferrous sulfate (copperas) and lime in place of the barium sulfide and lime when the chromium in the waste liquor was not in excess of from 75 to 100 p.p.m. Copperas was unable to precipitate greater amounts of chromium than this and in

such instances barium sulfide had to be used. In general, the use of copperas proved very satisfactory and eliminated many of the former difficulties.

When barium sulfide is used it is necessary to lower the pH of the raw liquor to 2.0 or 3.0 and then, after treatment, to bring the pH of the supernatant liquor back to 7.5 before discharging it. When copperas is used the pH of the raw liquor is raised to 8.0 by means of hydrated lime. For these reasons, and because of variations in the wastes themselves, careful pH control has been essential. Indicator paper did not suffice and conditions improved when a potentiometer was put into service.

New Treatment Method

In 1950 a new method of treatment was adopted which included sulfuric acid, sodium metabisulfite ($Na_2S_2O_5$), and hydrated lime. This appears to be the most satisfactory treatment of all. Whereas the barium sulfide is dark-colored, difficult to dissolve, and possessed of an unpleasant odor, the sodium metabisulfite is white, easily dissolved, and practically odorless.

As with barium sulfide, the pH of the raw liquor is lowered to 2.0 or 3.0 with sulfuric acid and then, after treatment, is raised to 7.5 by means of hydrated lime. The sodium metabisulfite dosage averages about 1.5 lb. per 1,000 gal., depending upon the concentration of the chromium in the raw wastes. It is said that the heavier this concentration is, the better the results. Although this chemical is slightly more expensive to purchase than barium sulfide, it is actually cheaper to use in the long run, due to its greater solubility and efficiency. Furthermore, the compounds formed as a result of this treatment are less objectionable than those produced when other reagents are used.

The sedimentation periods necessary when the different chemicals are used is a matter of interest. In a clean tank barium sulfide requires an average settling period of 2.5 hr.; in a tank in which the settled sludge is not removed, but is allowed to accumulate, it requires 4 hr. A satisfactory copperas treatment can only be obtained in a clean tank and the average settling period is 2 hr. The average settling period for sodium metabisulfite is 0.5 hr. when the tank is clean and 1.5 to 2 hr. when settled sludge is permitted to remain in the tank.

Sludge Disposal

At the outset a filter press was used to dewater the sludge. However, because this procedure was slow and also costly due to the amount of labor required, it was discontinued and a man who pumps out septic tanks was hired to cart the wet sludge from the settling tanks to a lagoon at the municipal sewage treatment plant. As this was done only once or twice a week, the sludge accumulated in the bottom of the settling tanks and was not conducive to good operation. In 1948 the company purchased a 1,500-gal. trailer tank, in which to transport the sludge to the disposal area; in 1950 it replaced this unit with a 3,000-gal. trailer tank. With this equipment the sludge can now be carted away as frequently as is found desirable.

Waste Quantity and Cost

At one time the volume of waste liquors ran up to nearly 0.5 m.g.d., but by making certain changes and by careful operation within the mill itself this volume was reduced to 0.25 m.g.d. or less per day, which is handled in the treatment plant over a period of two shifts.

The cost of the treatment plant was approximately $22,000 and cost of operation runs about $8,000 per year, including chemicals, power, labor, etc.

The successful operation of this plant is due primarily to the interest and conscientiousness of the plant engineer and the general maintenance foreman.

Metal Wastes Containing Cyanide

The problem at the American Screw Company, Willimantic, Conn., is one of treatment of cyanide-bearing waste liquors. In 1948, when this concern moved into the former Pratt and Whitney factory, it constructed a new industrial waste treatment plant designed for chemical treatment and sedimentation and consisting of a receiving well, two sets of pumps, three settling tanks, chlorine and caustic soda feeders, sludge-drying beds, and a combination treatment house and laboratory (see Figure 5).

The raw wastes enter the receiving well, from which they are pumped to the settling tanks. One set of pumps is manifolded to a pump suction header and discharges a portion of the wastes through the chlorinator ejector, where chlorine is added at a predetermined rate. The caustic soda is added to the pump suction before the wastes are pumped through the chlorinator ejector. This chemically-dosed portion is discharged to the influent header and to the particular tank being filled.

Provision is made for returning, through a so-called recirculation header, treated liquor from the settling tank being filled to the receiving well for the purpose of diluting the peak flows of cyanide and to smooth out the chlorine and caustic soda dosing rates. By this means it is also possible to reprocess an unsatisfactorily treated tankful of liquor, even while treating the incoming raw liquor entering another tank.

A continuous hydrogen ion concentration meter indicates and records the pH of the treated wastes. Because there is a tendency for the electrodes to become dirty and thus give an incorrect pH value, the meter is checked,

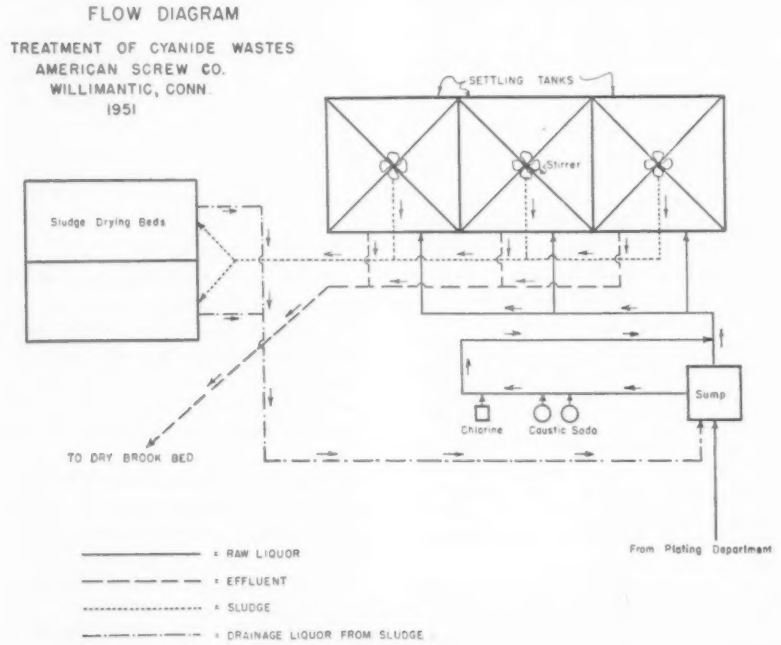

FIGURE 5.

and adjusted, if necessary, four times per shift by means of a colorimetric pH determination apparatus. There is a small oil separator ahead of the pH meter to protect it from such substances.

Average chemical dosage consists of approximately 40 to 45 lb. of chlorine per day and about 35 to 40 lb. of sodium hydroxide, although the latter can often be reduced due to the fact that the wastes at this factory are less acid than those discharged from the plating departments at most mills.

The caustic soda is dissolved in two steel solution tanks, each having a 120-gal. capacity and equipped with a lightning mixer. Feeding equipment consists of a variable capacity pump.

The chlorine is administered by a chlorinator and dosage is controlled by two rotameters.

There are three rectangular, concrete, hopper-bottomed settling tanks, each having a capacity of 22,000 gal. Mixing is accomplished quite effectively by means of a mechanism in the center of each tank consisting of a vertical shaft with a propeller at the bottom and driven from above at about 110 r.p.m.

It has been found that better results can be obtained if the treated liquor is stirred when the tank is half-full and again when it is full. Following the final stirring, the liquor is allowed to settle for at least 2 hr., depending upon the volume of waste reaching the treatment plant from the plating department. Before any tank is dumped the supernatant liquor is carefully tested. It must be clear, have a pH of 8.5 or more, and a chlorine residual of not less than 2 p.p.m.

Each settling tank is equipped with four 4-in. valved pipes, set at different elevations, by means of which the treated liquor may be decanted at the end of the sedimentation period. In actual practice the drawoff pipe nearest the bottom (that is, just above the hopper) is generally used. This liquor flows to a dry stream bed several hundred yards long, where considerable seepage into the ground takes place. That portion which does not go into the ground eventually reaches the Willimantic River, the flow of which provides adequate dilution for such an effluent.

The settled sludge is drawn off to sludge-drying beds through a 3-in. pipe set in the bottom of the hopper. The sludge is not drawn off at the end of each cycle, as it is considered advantageous in treating the wastes to permit these settled solids to accumulate in the settling tanks. Often two or three months elapse before sludge is transferred to the drying beds. There are two underdrained beds made of sand and gravel and having a total area of 900 sq. ft. The drained liquor flows back to the pump suction well. The dried sludge is so small in volume that its disposal has posed no problem.

Results and Costs

This treatment plant is effective in removing all of the cyanide from the waste plating liquors. From 80,000 to 90,000 g.p.d. of waste liquor are produced. Treatment is carried out during two 8-hr. shifts.

The treatment plant cost $50,000 to build and the annual operating cost runs about $7,000 or $8,000, most of which is for labor. However, the man on the day shift also operates the company's sewage disposal plant and the night operator does some work in the mill itself.

Paperboard Mill White Water

Several kinds of treatment systems have been used in Connecticut to handle the wastes from paperboard, fiberboard, and tissue mills. At the present time, however, sedimentation is most favored. This is particularly true since the major problem in Connecticut, because of the location of the mills, is one of solids removal rather than B.O.D. reduction. As experience has

indicated, plain sedimentation does little to remove or even reduce the dissolved B.O.D. of paper mill wastes.

At one fiberboard mill, where considerable air is introduced into the liquors during processing, an Adka filter has functioned most satisfactorily.

At a tissue mill an existing unit was converted into a plain settling tank with good results. By adding a little alum the suspended solids were reduced 98 per cent.

However, by far the most popular type of sedimentation unit appears to be the Marx conical save-all or a modification thereof. This unit (Figure 7) is actually a cone-shaped steel settling tank carried in an inverted position on steel legs. Inside the main cone is a slightly smaller truncated cone, also in an inverted position. In the upper part of the latter there is a slanting circular disc (or flat cone) surmounted by a conical receptacle about 3 or 4 ft. deep and having a much smaller diameter than the flat cone. The raw liquor drops into the center of the receptacle, flows over the edge and falls upon the slanting surface below. This is a de-aeration unit, the theory being that the impingement of the water upon the surface of the flat cone causes the entrained air to be liberated from the liquor, thus facilitating subsequent settling of the solids. As the liquor flows off the flat cone it passes downward through the inner cone, out through the open bottom of it and then upward between the inner and outer cones to the top of the latter, where it escapes over a weir extending around the entire periphery. The flowing-through period is generally 1.5 to 2 hr. From the weir a large proportion of the clarified liquor is returned to the mill for re-use and the remainder is discharged to the receiving stream. The solids settle in the bottom of the larger (outer) cone and are also pumped back into the mill for re-use.

Not only does such a system effect a saving in water and stock, but it also conserves heat units, as the returned liquor is usually warmer than fresh water from the stream. There is also some value to the dissolved

FIGURE 6.—Cyanide waste treatment plant at American Screw Co., Willimantic, Conn., showing concrete settling tanks, brick chemical treatment building, and sludge-drying beds (left foreground).

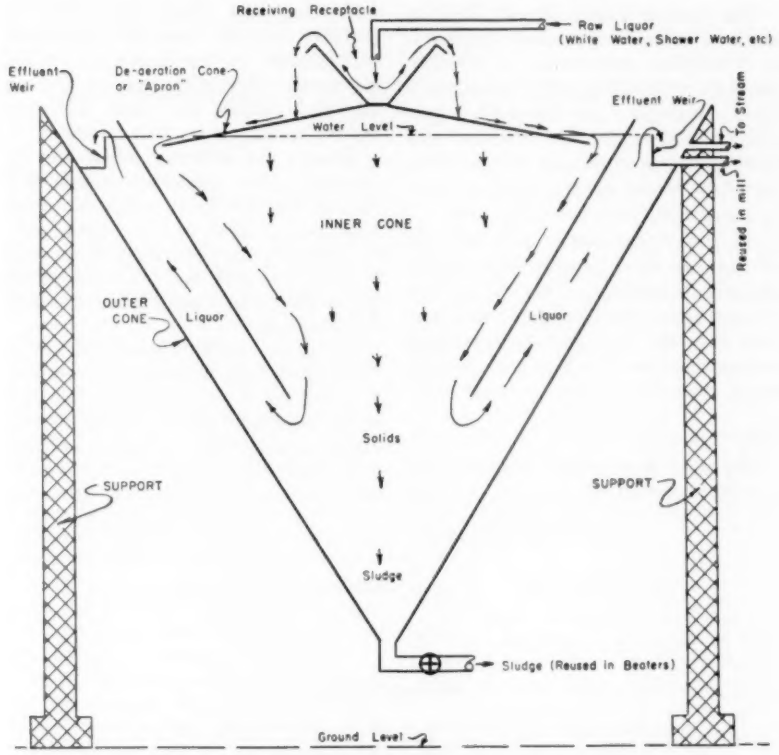

FIGURE 7.—Diagrammatic sketch of Marx conical save-all.

chemicals, which are thus returned to the manufacturing process.

pH Control Essential

To date the removal of suspended solids has varied from 82 to 95 per cent at the various mills. B.O.D. reduction is relatively low, due to the fact that it is mostly in solution and, therefore, not removable by sedimentation. Some of the mills have started experiments with the addition of alum to the settling tank influent in an effort to increase the removal of suspended solids. pH control is essential, as it has been found that the efficiency of the system decreases with a rise in pH, optimum conditions usually being obtained at about 4.5 to 4.8. Most of the mills neglected this in the past, but now realize the importance of such control.

Connecticut Installations

The first mill in Connecticut to install such a system was the National Folding Box Company, at New Haven, where a 96,000-gal. unit was erected. Later a 130,000-gal. unit was added to the system.

The New Haven Pulp and Board Company has also installed a 130,000-gal. unit with automatic pH control, and the Robertson Paper Box Company, at Montville, has a 79,000-gal. unit in operation.

The Inland Paper Board Division of the Federal Paperboard Company, at Versailles, constructed a 200,000-gal. concrete settling tank based on the Marx principle. At this mill a mechanically-cleaned trash rack and skip hoist and two vibrating screens were installed ahead of the settling tank to remove all extraneous material.

All progressive paper mills now recirculate as much process water within the mill as possible. This reduces the volume ultimately discharged and permits the use of a smaller treatment system than would otherwise be necessary.

Costs

The total installation cost of these systems has varied, according to size and complexity, from approximately $50,000 for the smallest mentioned to $150,000 for the largest, including all items. Operating costs vary from $2,000 to $6,000 per year, including power, maintenance, and labor.

This is one industry where the industrial waste treatment system will usually pay for itself within a reasonable period of time. In most cases the value of stock recovered, water and chemicals re-used, and heat units saved more than compensates for the cost of operation.

All of the projects described are illustrations that interest and cooperation on the part of factory management, treatment plant personnel, and state agencies will produce satisfactory results when solutions are sought for industrial waste disposal problems.

CORRECTION

In the paper on "Evaluation of Methods for Measurement of Free Cyanide," by E. G. Will (This Journal, 23, 10, 1288; Oct., 1951), in the last paragraph under the heading "Phenolphthalin Method" (page 1289) the word "phenolphthalin" should read "phenolphthalein."

DEMONSTRATION PLANT FOR TREATMENT OF DE-INKING WASTE *

By A. J. Palladino

Engineer, National Council for Stream Improvement

A treatment plant to demonstrate the feasibility on a full-scale basis of biological aeration of de-inking waste water from the paper industry began operation in September, 1950. This plant was constructed at the Michigan Paper Company, Plainwell, Mich. by the Kalamazoo River Improvement Co., consisting of six "book" mills in the Kalamazoo area which have in common the problem of disposal of de-inking waste. Design and supervision of construction and operation were delegated to engineers of the National Council for Stream Improvement. The design was based on previous laboratory development by the National Council and pilot plant tests conducted cooperatively with the industry, which had indicated the effectiveness of this process from a treatment efficiency standpoint. The demonstration plant operation, however, was directed toward compiling information relative to full-scale operation—that is, maintenance problems, sludge disposal requirements, and cost data, as well as confirmatory data regarding treatment efficiencies and design criteria.

According to the terms of the agreement by which the Kalamazoo River Improvement Company was formed, this plant is to be operated for a one-year period, following which the data will be compiled and the over-all feasibility assessed. Therefore, no operating data are presented herein. This paper presents the design features of the plant, the operating techniques, and the aids which are used to evaluate operating conditions.

In regard to the nature of de-inking waste (which is discharged from the pulp washers following alkaline cooking of old paper) and experimental treatment methods, considerable literature is available (1)(2)(3)(4). Also, the design of this demonstration plant has been detailed in previous publications (5). Average characteristics of waste treated at Plainwell are presented in Table I.

Design Features

The plant (Figure 1) consists of a primary settling tank, aeration tank, secondary settling tank, secondary sludge concentrating tank, primary and secondary sludge-drying lagoons and a wooden frame control building. All units stand above ground. The primary units were designed for a flow of 0.5 m.g.d. Design flow through the secondary units was 0.17 m.g.d., but allowance was made for adjustment of the secondary flow to any desired percentage of 0.5 m.g.d. Unit capacities are presented in Table II.

The de-inking waste is piped from the source to a large collection tank in the mill basement. This tank is equipped with an overflow weir to per-

TABLE I.—Average Characteristics of De-Inking Waste as Received at Plant

Total solids (p.p.m.)	3,700
Volatile (%)	46
Suspended solids (p.p.m.)	2,200
Volatile (%)	30
B.O.D. (p.p.m.)	400
pH	8.3
Temperature (° F.)	80

* Presented at 1951 Annual Meeting, Michigan Sewage and Industrial Wastes Association; Grand Rapids, Mich.; June 4–6, 1951.

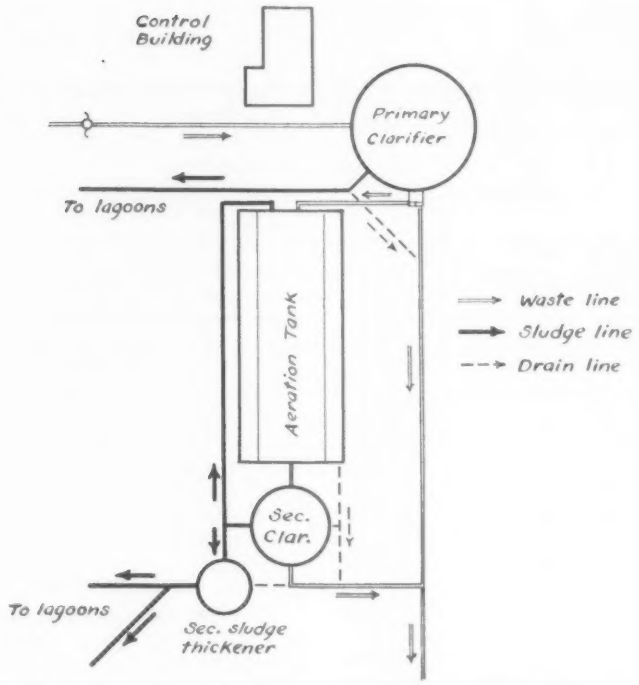

FIGURE 1.—Flow diagram of demonstration plant for de-inking waste treatment.

mit measurement of total waste flow, inasmuch as the total flow is not pumped to the treatment plant. Subsequent to pumping into the primary tank the flow through the plant is by gravity. All operations except sludge removal are automatic. Sludge is pumped by a screw-type positive displacement pump from the primary tank to the sludge-drying lagoons.

The sludge averages about 10 per cent solids, but will occasionally reach

TABLE II.—Basic Design Data for Demonstration Plant Treating De-Inking Wastes

Unit	Diameter (ft.)	Length (ft.)	Width (ft.)	Water Depth (ft.)	Capacity (cu. ft.)	Detention Time (hr.)[1]
Primary sett. tank	26	—	—	10	5,300	2
Aeration tank	—	52	14–21	11	11,000	8[2]
Secondary sett. tank	16	—	—	9	1,800	2
Sludge conc. tank	16	[3]	[3]	10	700	15
Lagoons:						
Primary	—	—	—	3	72,700	90[4]
Secondary	—	—	—	3	18,000	90[4]

[1] For primary flow of 0.50 m.g.d.; for secondary flow of 0.17 m.g.d.
[2] Allows for return sludge flow of 0.083 m.g.d.
[3] Hopper bottom.
[4] Days.

15 per cent. Waste secondary sludge is discharged from the concentration tank to the secondary sludge lagoons by hydrostatic pressure. This sludge (normally about 2 per cent solids) can be concentrated overnight to 10 per cent solids.

The primary tank and aeration tank are of steel construction, the latter being supported directly on a 24-in. gravel bed. The combination of tar undercoating and good drainage is expected to prevent undue corrosion of the aeration tank bottom. Other unique features of the aeration tank are:

1. Semi-elliptical cross-section; that is, flat bottom and round sides. Steel construction lent itself to such design in lieu of the conventional vertical side-walls with top and bottom fillets. The circulation in the tank is apparently excellent.

2. By loosening a union, the air headers can be swung out of the tank to the opposite side for cleaning. Since the diffusers (which consist of ¾-in. pipe with ⅛-in. opening) must be cleaned once a week, it is important that the headers be removed with facility.

The secondary settling tank and the sludge concentrating tank are constructed of wood, inasmuch as they were considered to be of a temporary nature. Return secondary sludge is lifted by means of an air lift to a weir box atop the secondary settling tank for measurement and is then returned by gravity to the aeration tank. The return sludge ratio is about 40 per cent.

Some of the above-mentioned features are apparent in Figures 2, 3, and 4. The entire plant was designed with a view to facilitating possible future expansion.

Flow Measurement

Waste Water

The waste-water flow is measured in a weir box, which was made an integral part of the primary tank overflow system. Two adjustable weir plates split the primary tank overflow in any desired ratio into two portions; one is directed through the secondary system, the other is discharged directly to the plant effluent sewer. The weir plates are so set in elevation as to allow no free fall from the tank launder into the weir box, thus minimizing head loss and turbulence in the weir box. A continuous record of the heads is obtained by means of a float recorder. Thus,

FIGURE 2.—Demonstration de-inking waste treatment plant, showing primary clarifier (left); aeration tank (right center); and secondary sludge concentration tank (extreme right). Secondary settling tank is behind right end of aeration tank.

two flows are computed by means of a single head record.

Sludge

Measurement of the volume of primary sludge withdrawn is accomplished by noting the head discrepancy in the primary tank overflow record during the sludge-pumping period. For greater accuracy, the latter is recorded rather than taken from the charts.

Secondary sludge discharged to and wasted from the concentrating tank is measured by noting the volume displacement in the tank. A chart relating tank depth to volume is available for this purpose.

Air Flow

Total air output by the blower (of the multiple centrifugal type) and the flow to each header are measured by means of orifice plates placed in the 6-in. air main upstream and downstream of each header takeoff. Air flow averages about 4 cu. ft. per gallon of waste treated.

Backpressure in each header is also measured. The combination of air flow and backpressure measurements serves as an index to the time for diffuser cleaning. Air flow is markedly affected by variations in backpressure.

Operating Details

Exclusive of short shut-downs at the mill, waste is received at the plant continuously every day but Sunday. The flow is practically constant at 0.50 m.g.d. through the primary unit. Current flow through the secondary units is constant at 0.20 m.g.d., but is to be increased by increments to determine the ultimate allowable loading. One man working only the day shift operates and maintains the plant, runs the

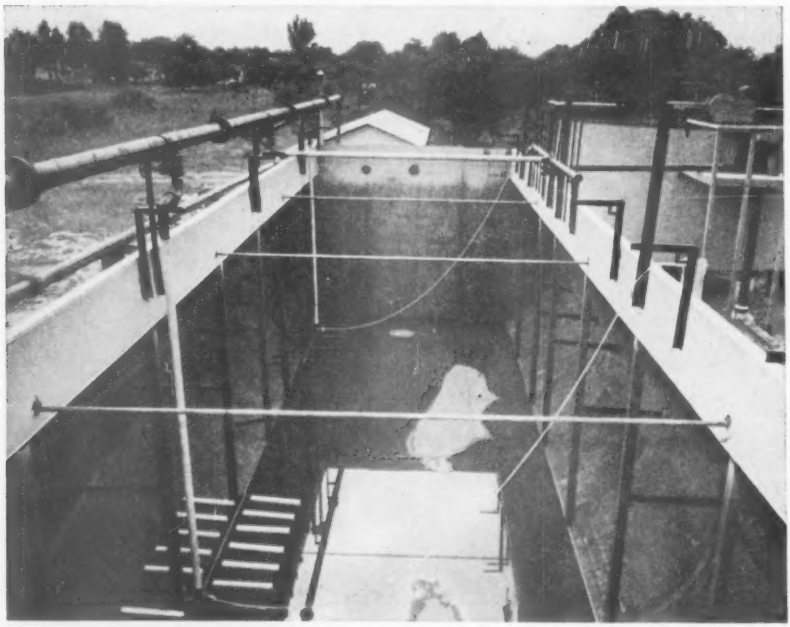

FIGURE 3.—Inside view of empty aeration tank, showing return sludge and raw wastes inlets (far end) and method of raising air diffusers.

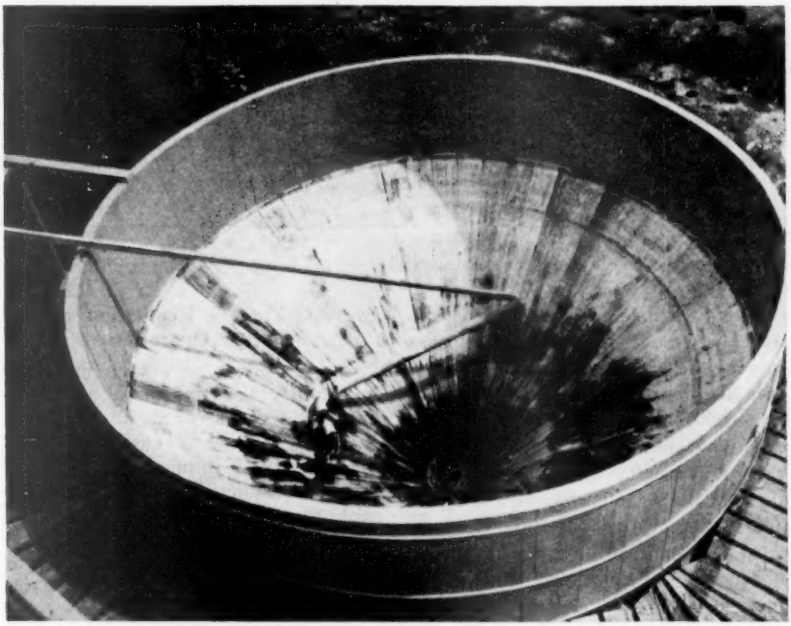

FIGURE 4.—Secondary sludge concentration tank, showing manually adjustable supernatant removal line.

control tests, and keeps the plant records. Outside of time required for maintenance, the operator is concerned essentially with the control tests and record keeping. The control tests and schedule are shown in Table III.

Spot, rather than composite samples are taken for analysis. Composite samples would be of greatest value on primary influent samples. Effluent samples vary to a much lesser extent, so that spot samples are fairly representative. Over a considerable period of time, the average of spot sample analysis is probably as valid as the average of composite sample analyses. In addition to these tests, periodic checks of the solids and volatile content of all sludges are made.

With few exceptions sludge is wasted daily from the primary and secondary operations. A sludge depth "feeler" and sludge consistency are utilized as indices to the primary sludge removal cycle. This sludge is removed slowly and continuously over a period of 6 to 10 hr. at a rate of about 20 g.p.m. Secondary sludge is wasted in amounts sufficient to maintain the mixed liquor concentration at about 5,000 p.p.m. Once this level is attained the following formula is used as a guide to the amount of sludge which should be wasted—

$$\text{Waste Secondary Sludge (gal.)} = \frac{100 \times \text{flow (m.g.d.)} \times [(a-b)+(c-d)]}{\%\ \text{solids in secondary sludge}}$$

—in which a represents the p.p.m. of suspended solids in the secondary influent; b, the p.p.m. of suspended solids in the secondary effluent; c, the p.p.m. of dissolved solids in the secondary influent; and d, the dissolved solids in the secondary effluent.

TABLE III.—Types and Schedule of Control Tests

Location	Total Solids[1]		Sus. Solids		Sett. Solids	Sludge Volume Index	B.O.D.	D.O.[2]	pH[2]	Temp.[2]
	Tot.	Vol.	Tot.	Vol.						
Primary infl.	x	x	x	x	x	—	x	—	x	x
Primary effl.	—	—	x	x	x	—	x	—	x	x
Aeration tank	—	—	x	x	x	x	—	x	x	x
Final sett. tank	x	x	x	x	x	—	x	x	x	x

[1] Every other day.
[2] Occasional tests. All other tests made daily.

This formula represents a solids balance between that entering and that leaving the secondary system. It takes into account the conversion of soluble material to insoluble material in the aeration tank. Average results indicate a reduction of soluble solids through the secondary system of about 300 p.p.m. Much of this decrease must be accounted for by a corresponding increase in suspended solids in the form of microbiological growth. Frequently, suspended solids in the secondary influent are lower than in the secondary effluent, so that the first part of the solids factor is negative.

Sodium nitrate (12 p.p.m.) and kerosene (1:200,000) are added to the flow directed through the secondary system. The nitrate acts as a nutrient for biological growth, whereas the kerosene is used to prevent excessive foaming in the aeration tank.

Experimentation with commercial sludge dewatering equipment and air diffusers is being conducted.

Aids to the Evaluation of Plant Operation

Several charts have been made up to simplify the evaluation and compilation of operating data. These include weir discharge curves, air flow charts, tank volume displacement charts, and sludge lagoon depth-volume calibration curves. In regard to the latter, calibrated 2×4's standing in the sludge lagoons serve as an index to sludge depth, which can in turn be converted to compacted sludge volume by means of the volume calibration curve. A continuous comparison between the compacted sludge volume and the cumulative volume of uncompacted sludge added to the lagoon is thus available.

Record Keeping

All analyses are recorded in bound data books and transcribed on mimeographed data sheets. These have been designed to facilitate the compilation and interpretation of the data. A daily log is also maintained for this purpose.

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OPERATION OF A MILK WASTES TREATMENT PLANT EMPLOYING A TRICKLING FILTER*

By John W. Rugaber

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In 1937, the Pet Milk Company erected an evaporated milk plant at Coldwater, Ohio. This was a completely new installation; the old plant was abandoned except for use as a maintenance shop and garage. Included in the new construction was a complete trickling filter system for treating the industrial wastes from the the milk plant operations. The trickling filter plant was designed by H. P. Jones and Company, consulting engineers, Toledo, Ohio, and the plans were approved by the Ohio Department of Health on April 30, 1937.

The wastes to be handled consist primarily of milky waste water, resulting from emptying and washing farmers' milk cans in the receiving room, the daily washing of all processing equipment, and the washing of the stainless steel pipelines and storage tanks. There is also some waste from cleanup operations in the ice cream mix department. The waste from the last source contains sugar as well as milk. The sanitary sewage from the rest rooms goes to a standard septic tank; however, the effluent from the septic tank joins the influent to the trickling filter plant.

The original installation (Figure 1) consisted of a small brick building containing pumps and sumps, together with the necessary control equipment; a concrete storage tank; a trickling filter; and a final settling tank.

* Presented at 1951 Annual Meeting, Ohio Sewage and Industrial Wastes Treatment Conference; Columbus, Ohio; June 20–22, 1951.

Flow Variations

The milk plant was designed to handle 300,000 lb. of whole milk per day. The major part of the processing and about half of the cleanup work was done in a 10-hr. day, with the heavy cleanup work extending an additional 3 or 4 hr. at certain times of the year.

Due to the waste being produced only a few hours a day, it was decided to accumulate part of the waste in a holding tank during the milk plant operating hours, and then feed this onto the trickling filter at night. This was done to put a lower load on the filter during the day and to feed the filter growth at night. The arrangement reduced the size of the trickling filter which would have been required for peak loads and provided a more balanced daily operation.

The waste entering the pump house goes into a primary settling tank with two outlets. From one of the outlets an adjustable orifice feeds part of the waste to the head box of the filter pump. When the influent exceeds the flow permitted through the orifice, a float switch controls a pump to pump the excess to the storage tank. Whenever the flow of waste from the milk plant is less than the desired flow set by the orifice to the head box, a float valve opens and permits waste from the storage tank to enter the system.

The original design of the trickling filter provided for an applied waste of 200 p.p.m. B.O.D. To reduce the waste to this strength, a float valve feeds sewage plant effluent for dilution water to the head box and the orifice is set to feed the amount of raw waste

FIGURE 1.—Waste treatment plant of Pet Milk Co., Coldwater, Ohio. Concrete tank in foreground, originally built as holding tank to level out flow to trickling filter, is now used as aerator to prevent septicity of held wastes.

permissible to maintain the desired strength.

The trickling filter is of concrete construction, 87 ft. inside diameter by 10 ft. deep, and with an 8-ft. depth of filter slag. The final tank is 22 ft. in diameter by 5 ft. effective depth, containing 14,250 gal. As the pump handles 140 g.p.m., the nominal detention period is 1.7 hr.

The original design called for pumping liquid sludge from the primary and final settling tanks into a tank truck to be hauled away.

The plant was placed in operation as described, and the first record of the results of operation is from tests made by the Ohio Department of Health in 1939.

The 1939 investigations showed an average over-all plant efficiency based on B.O.D. removal, of 98.3 per cent. The pH value of the raw waste in 1939 averaged 7.8, although the variation in samples was from 6.2 to 9.6. Due to the large amount of dilution by recirculation, the trickling filter probably was not exposed to a wide variation of pH. Milk rapidly forms lactic acid; but the alkaline cleaning compounds used in the plant cleanup work neutralize the lactic acid fairly well in most plants.

Clogged Filter

This is all the information available on the pre-war design and operation of the Coldwater plant. Unfortunately the waste treatment plant suffered seriously from the loss of personnel during the war years. In the spring of 1946 the author was sent to Coldwater to clean up the filter plant and try to correct certain operating conditions.

The trickling filter had become badly loaded with sludge. The waste was ponding and channeling, and sludge was piled up for lack of disposal facilities. Hauling the sludge away in trucks, as originally planned, had stopped due to the lack of a place to haul it. The original plan had been to dump the sludge on farm land. However, farmers would not accept such materials during the growing season, and truck operation on soft ground at certain times of the year was practically impossible. It had, therefore, become necessary to discontinue hauling the liquid sludge away in tank trucks; the resulting accumulation of sludge throughout the plant had greatly reduced the operating efficiency.

Three sludge-drying beds had been

built, but were very much overloaded. The practice had been to pump from the primary and final tanks daily, using the same bed for a week, then let the bed dry for a week, clean the bed the third week, and start over on the fourth week. When the first day's sludge partially dried, a surface developed through which liquid could not penetrate. When the second day's sludge went on top of the first, the only escape for the liquid was through the drying cracks in the previous day's sludge. Each day the area of these channels became less; by the end of the week the beds could not drain, they simply had to air-dry from the surface. In wet weather, drying was impossible.

Change in Drying Beds

The three existing beds, after being cleaned, were divided into sections (Figure 2) so that two layers of sludge never went on the same bed. Sludge was applied deeper, but to a smaller area and all in one continuous layer on each section. As a result of this change, it was possible to dry the sludge reasonably well in a week's time, except for very bad weather.

Arrangements having been made to haul the accumulated sludge away, various methods were tried to make the filter unload, but without success. Finally it was necessary to take on the unsavory job of removing the filter slag and washing it. A power shovel was hired to handle the slag, and the city of Coldwater very kindly loaned a fire engine pumper and hoses for doing the washing. The underdrains were almost completely plugged and the sludge had to be cleaned from these drains also.

After thoroughly cleaning the system, it was returned to operation with only fair success. Everything worked fine during the day, but when the storage tank was used troubles developed again.

Aeration of Wastes

By this time, interest had been aroused in aeration and it was decided to try out an addition to the original system. A gear-type blower, which was on hand, was installed in the pump

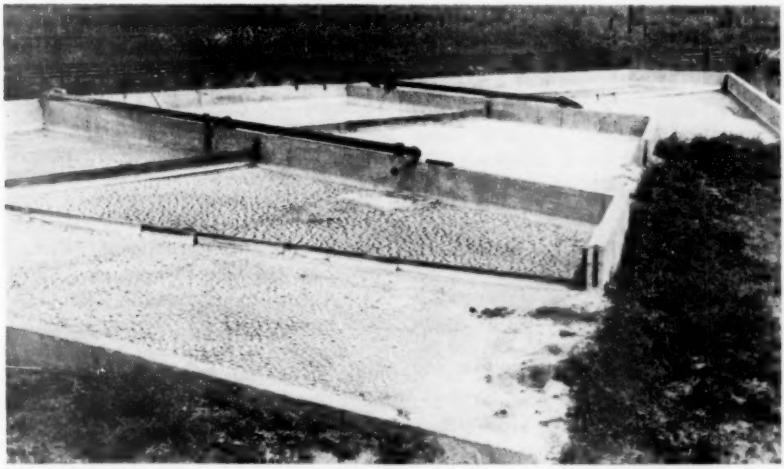

FIGURE 2.—Wooden dams divide sludge-drying beds into smaller areas to reduce drying time.

house and an air distributing pipe was installed in the holding tank. A small amount of air was also introduced at the pump head box, but the storage tank was the chief concern.

It had been noticed that the waste in the storage tank rapidly became septic, and there had been increasing sludge problems when the storage tank was used. The air equipment available did not appreciably reduce the B.O.D. in the storage tank, but did keep the storage tank waste in as good or better condition than when originally received. Odors were reduced and the sludge problems were very much improved. The over-all treatment plant operation reacted very favorably to the aeration operation.

Treatment plant operation continued to improve during the remainder of the year and by 1947 it was felt that operation was reasonably satisfactory. During the heavy milk production period of 1950, the routine testing of samples from the filter plant indicated from 95 to 99 per cent over-all plant efficiency based on B.O.D. removal. Raw waste strength varied from 560 to 2,880 and the final effluent from 5 to 54 p.p.m. B.O.D. Average raw waste strength was 1,830 and the average final effluent was 25 p.p.m. B.O.D.

The final effluent is now being recirculated on about a 5 to 1 ratio.

As a result of experience with this plant, the important points in satisfactory operation are as follows:

1. Do not allow the waste to become septic before processing it through the trickling filter.
2. The use of a small amount of air can prevent septic conditions when storage is necessary.
3. Keep sludge pumped out of all settling tanks daily, and clean the storage tank daily.

If a new filter were being built today, it would be considered necessary to change the design of the underdrains. It would also be desirable to try forced air circulation through the trickling filter system.

Plant with Enclosed Filter

This concludes the report on the Coldwater operation. However, it would be of interest to mention briefly the experience with another type of trickling filter plant.

This plant consisted of a primary settling tank, a trickling filter, and a final settling tank. Sludge was pumped into a septic tank and then hauled away annually. The filter had been enclosed in a concrete block building with fair ventilation. State health department tests showed 92 per cent efficiency on B.O.D. removal. However, this is one good example of how little efficiency can really mean; the effluent still had 500 p.p.m. B.O.D. The influent to this plant contained 5,700 p.p.m. B.O.D., due to lack of a waste-saving program in the plant and very little dilution water.

The enclosed filter, operating with no exposure to sunlight and very little light of any kind, does not seem to develop the type of growth customarily found on trickling filters.

Conclusion

The results at Coldwater have proven beyond any doubt that a trickling filter plant, properly designed and properly operated, can do an excellent job of milk waste treatment. Unfortunately, the cost of construction of such a plant today, including the taxation involved on the increased capital investment, is more than many small milk plants can bear. The trickling filter plant certainly is not an economical answer to the milk waste disposal problem.

DESIGN AND OPERATION OF A METAL WASTES TREATMENT PLANT *

By C. M. Gard, C. A. Snavely, and D. J. Lemon

Respectively, Sanitary Engineer, Alden E. Stilson and Associates; Assistant Division Supervisor, Battelle Memorial Institute; and Sanitary Engineer, Ranco Inc.

The Delaware, Ohio, plant of Ranco Inc. has produced automobile heater controls since starting operations in April, 1948. The production methods in general follow those developed from previous experimentation and small-scale production at the main plant in Columbus, Ohio.

The cleaning and plating operations involved include oil and dirt removal from stampings by washing with an alkaline cleaner, cleaning and acid dips in preparation for plating, zinc and copper plating, and Iridite treatment (chromating) of certain parts. The zinc and copper-plating solutions are of the cyanide type. The volume of copper plating is relatively small. The volume of zinc plating is substantial and is handled by two-shift operation of 10 barrel-type units of 600-amp. capacity.

Both the plating wastes and the sanitary wastes from the plant were originally discharged through a single 8-in. sewer to the city sanitary sewer at the edge of the Ranco property. All storm waters were collected separately and discharged into a natural drainage course.

City Sewage Treatment Plant

The Delaware sewage treatment plant is of the conventional trickling filter type, consisting of a bar screen, wet well, raw sewage pumps, Imhoff tanks, trickling filter with fixed nozzles, final settling tank, and sludge-drying beds. Because of inadequate capacity, the final settling tank is not used. The filter effluent is, therefore, the final effluent and is discharged into the Olentangy River.

In 1949, about 16 months after the start of operations at the new Ranco plant, the city made formal complaints to the Ranco management regarding the character of the plant wastes being discharged to the municipal sewer system. It was claimed that these wastes were having deleterious effects on the sewer system equipment and on the treatment process itself.

The industry took immediate action. The firm of Alden E. Stilson & Associates of Columbus, Ohio, was engaged to survey the waste problem and recommend a course of action. Battelle Memorial Institute of Columbus, Ohio, assisted in carrying out the survey and preparing recommendations for a treatment process.

The Waste Survey

The survey schedule was designed to determine the quantity and chemical and physical characteristics of the wastes. It was conducted over a consecutive five-day period while the plant was in full three-shift operation. A special sampling box was installed in the 8-in. sewer leading from the plant. A 90° V-notch weir in this box provided means for flow measurements which were continuously recorded. A special proportional sampling device was constructed and installed ahead of the weir plate. This sampler withdrew

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a sample of the flowing waste every two minutes in direct proportion to the momentary rate of flow. These samples were collected in 5-gal. Pyrex carboys so that a composited sample, weighted in proportion to the flow, was obtained for each calendar day.

The pH of the waste was measured at ½-hr. intervals at the sampling box and at a sump in the main plating room. The major portion of the cleaning and plating wastes passed through this sump.

During the first three days of the survey period, the wastes were from normal factory operations. During the last two days, no processing tank contents were dumped. Thus, the contaminants during these last two days were limited to those contained in solution dragged out of the processing tanks and appearing in the rinse waters only. There was no significant change in the chemical character of the wastes during the last two days.

A summary of the wastes analyses obtained in this survey is given in Table I. The data are for 24-hr. composite samples. Although it was known that cyanides would be present in the wastes, no data were obtained on the amounts. It was decided at the start of the survey that analyses for cyanides would be no value, because an indeterminant amount would be broken down by the free acid present.

The pH measurements taken at the sampler box for the period of the survey are plotted in Figure 1. These pH measurements were made with indicator papers and, therefore, are not precise. They are indicative, however, of the hourly variations. Other pH measurements in the survey were made with a glass electrode meter.

A concurrent sampling program was conducted at the city sewage treatment plant. Samples of the raw sewage and the final effluent were collected every 2 hr. and the pH measured. These samples were then composited for each 24-hr. period in proportion to the sewage flow. Complete physical and chem-

TABLE I.—Analysis of Ranco Plant Wastes during 5-Day Survey; 24-Hr. Composite Samples

Item	Mon.	Tues.	Wed.	Thurs.	Fri.	Av.
Total flow[1] (m.g.d.)	0.299	0.332	0.351	0.368	0.362	0.342
Total solids (p.p.m.)	828	833	756	648	680	749
Volatile (p.p.m.)	188	215	155	156	158	175
Suspended solids (p.p.m.)	32	37	31	18	29	29
Volatile (p.p.m.)	29	35	19	15	25	25
Dissolved solids (p.p.m.)	798	796	728	631	650	720
Volatile (p.p.m.)	160	182	136	144	133	150
Acidity, as H_2SO_4 (p.p.m.)	466	489	436	382	344	424
Sulfates (p.p.m.)	507	495	463	405	370	448
pH	1.5	1.5	1.5	1.6	1.7	1.6
Copper (p.p.m.)	28	33	29	26	24	28
Chromium, total (p.p.m.)	39	41	32	26	30	34
Hexavalent (p.p.m.)	25	23	19	15	19	20
Trivalent (p.p.m.)	14	18	13	11	11	14
Zinc (p.p.m.)	15	20	15	14	15	16
Cadmium (p.p.m.)	14	22	—	—	—	18
Nickel (p.p.m.)	1.1	0.6	—	—	—	0.9
5-day B.O.D.[2] (p.p.m.)	68	56	62	29	40	51
Petr. ether sol. (p.p.m.)	6.2	—	—	—	—	6.2
$Ca(OH)_2$, pH 9.0[3] (lb./m.g.)	2,090	—	—	1,670	—	1,880

[1] Total industrial and sanitary flow.
[2] Not adjusted for toxic effect of metal ions.
[3] Amount of $Ca(OH)_2$ needed to raise pH of wastes to 9.0.

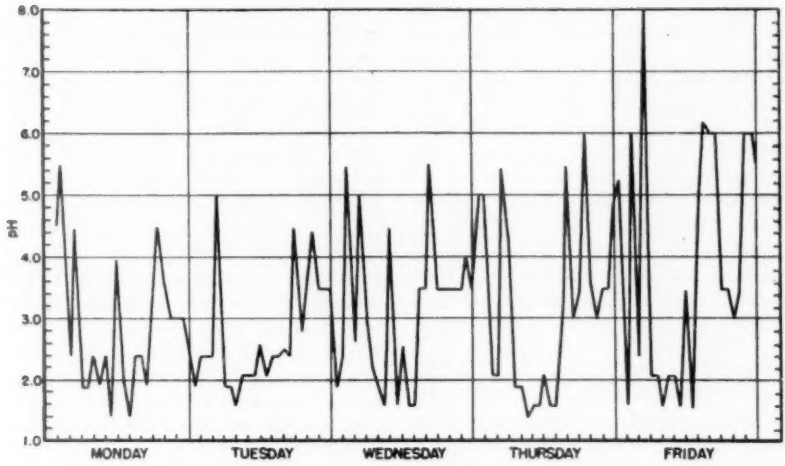

FIGURE 1.—Variations in pH of waste flow during survey period.

ical analyses were made on each of these daily composited samples.

Treatment Requirements

The city of Delaware, Ohio, established the following standards of quality for wastes entering the city sewer system:

pH	6.5 to 9.5
Total Fe	5 p.p.m. or less
Cu	1 p.p.m. or less
Cr	3 p.p.m. or less
CN	None
Mineral oil	None
Soluble oil	20 p.p.m. or maximum practical removal

Sand, abrasives, metal turnings, etc., eliminated to the greatest practical extent.

The consulting engineers were authorized by the Ranco management to select and prepare plans for a treatment process that would produce a treated waste meeting the city standards.

Process Design

Substantially complete treatment was indicated for the removal of the toxic metals and cyanides, the neutralization of free acids, and the removal of oils.

The presence of sanitary sewage with the industrial wastes would complicate the handling and treatment of the wastes, so that segregation of the wastes within the Ranco plant was considered necessary. All sanitary sewage would be collected separately and discharged without treatment directly into the city sewers. The industrial wastes would be collected separately and further segregated to meet the treatment process required.

After a thorough review of the many methods developed for the treatment of cyanides, it was decided that the oxidation of the cyanides by the addition of chlorine would be the most practical (1). According to Dobson (2), simple cyanide salts are oxidized by chlorine in two stages, first to cyanates, then to carbon dioxide and nitrogen, according to the following equations:

$$NaCN + 2NaOH + Cl_2 \rightarrow NaCNO + 2NaCl + H_2O \quad (1)$$

$$2NaCNO + 4NaOH + 3Cl_2 \rightarrow 2CO_2 + 6NaCl + N_2 + 2H_2O \quad (2)$$

The stoichiometric quantities are 2.73 lb. of chlorine per pound of cyanide

(CN) to convert to cyanates as in Eq. 1, and 4.09 lb. of chlorine per pound of cyanide (CN) additional to break down the cyanates as in Eq. 2. The minimum pH recommended was 8.5. This required that cyanide and other alkaline wastes be collected separately from acid wastes.

The toxic metals present, such as chromium, copper, and zinc, can be removed as insoluble hydroxides in alkaline solutions. Chromium, however, is more difficult to remove than the other metals. All chromium must be in the trivalent form to be so precipitated. Because about one-half of the total chromium present was in the hexavalent form, provisions had to be made to reduce this hexavalent chromium.

The use of sodium metabisulfite ($Na_2S_2O_5$) was selected as the reducing agent. Commercially, this chemical is known as A.B.S. (anhydrous bisulfite). Its selection was based on its availability, its ease of handling in the dry form, and the smaller quantities of sludge produced as compared with such reducing agents as ferrous sulfate. The bisulfite costs more per pound than ferrous sulfate, but less is required on a theoretical basis.

The reaction between sodium metabisulfite and chromates and dichromates is illustrated by the following:

$$3Na_2S_2O_5 + 4Na_2CrO_4 + 10H_2SO_4 \rightarrow 2Cr_2(SO_4)_3 + 4Na_2SO_4 + 6NaHSO_4 + 10H_2O \quad (3)$$

$$3Na_2S_2O_5 + 2Na_2Cr_2O_7 + 8H_2SO_4 + 3H_2O \rightarrow 2Cr_2(SO_4)_3 + 2Na_2SO_4 + 6NaHSO_4 + 8H_2O \quad (4)$$

Theoretically, it requires 2.81 lb. of sodium metabisulfite to reduce 1 lb. of hexavalent chromium and no precipitate is formed. In comparison, it requires 16 lb. of ferrous sulfate per pound of hexavalent chromium reduced, and 6.15 lb. of ferric hydroxide is precipitated as sludge.

The efficiency of reducing chromium is dependent upon the pH of the solution. At a pH of 1 to 1.5 complete reduction to the trivalent state can be obtained with close to the theoretical amounts of reducing agent. As the pH increases above 1.5, excess reducing agent is required. A pH of 4.0 is too high for practical purposes where complete chromium removal is desired. These facts dictated that all chromium and acid wastes be collected together for preliminary treatment before mixing the alkaline and acid wastes for final pH adjustment. There was some possibility that pH adjustment of the acid waste might be necessary for the most efficient chromium reduction.

Because of the relatively small amounts of oily material present in the wastes, it was not considered necessary to segregate them for separate treatment. It was believed that the quantities involved could be sufficiently entrained by the sludge produced in the precipitation of the metallic hydroxides.

The final segregation of the plant wastes was established as follows:

1. All sanitary wastes would go directly to the city sewers without treatment.
2. All industrial wastes would be treated prior to discharge to the city sewers.
3. The industrial wastes would be further segregated into (a) all acid wastes including chromium, and (b) all alkaline wastes including cyanides, cleaners, and small quantities of oils.

In summary, the basic features for the treatment of the wastes were developed as follows:

1. Segregation of wastes as previously outlined.
2. Treatment of the cyanide-bearing alkaline wastes with chlorine to oxidize the cyanides either to cyanates or to complete breakdown.

3. Treatment of the chromium-bearing acid wastes with sodium metabisulfite to reduce all chromium to the trivalent form.

4. Mixing of the pretreated acid and alkaline wastes with the addition of sufficient lime to produce the optimum pH so that the free acids would be neutralized and all metals precipitated as hydroxides.

5. Discharge of the clarified liquor to the city sewers and discharge of the sludge with entrained oils to lagoons.

Plant Design

All wastes are segregated and conducted by gravity to sumps located throughout the factory area. Each sump is provided with float-controlled pumps for conducting the wastes to the treatment plant.

The survey report carried the recommendation that the quantity of rinse waters used in the metal-finishing operations be substantially reduced. The recommended changes and others developed by Ranco Inc. resulted in an over-all reduction of about 40 per cent in the amount of water used in the factory. The final design flows were based on the reduced water consumption resulting from these operational changes.

The treatment plant is designed to handle 100 g.p.m each for the acid and alkaline wastes, or for a total combined flow of 200 g.p.m. Because of the large quantities of flow, a continuous treatment process was adopted in preference to a batch type. This effected considerable savings in the number and size of treatment tanks required. Figure 2 shows a schematic flow diagram of the treatment plant.

Two 18,000-gal. holding tanks are provided for receiving the raw wastes as pumped from the factory area. One tank is for the acid wastes, the other for the alkaline wastes. Mechanical mixers in each tank provide about 15 turnovers per hour.

Treatment Tanks

From the alkaline holding tank, the alkaline-cyanide wastes go by gravity through an automatic rate controller and flow meter to two tanks for oxidation of the cyanides. Each tank has a capacity of 3,000 gal. and the piping is arranged so that either tank can be

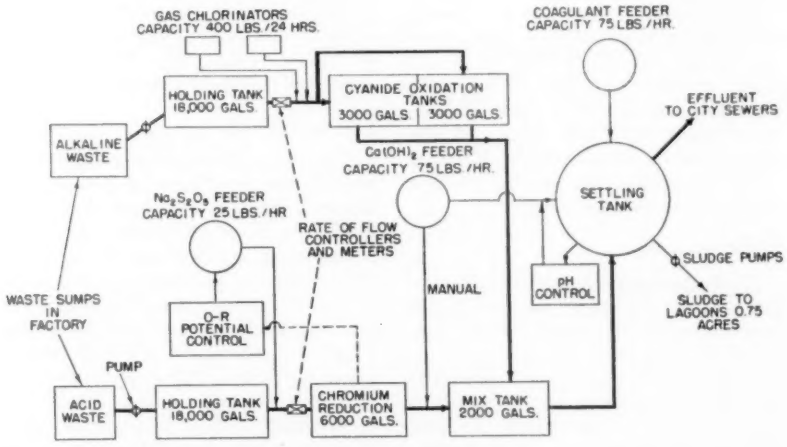

FIGURE 2.—Schematic flow diagram of waste treatment plant.

used alone or both can be used in series. The total detention time for the tanks in series is 1 hr. at the design flow of 100 g.p.m. Mechanical mixers installed in each tank provide about 126 turnovers per hour. Chlorine can be applied at the inlet line of either tank. The chlorine can be fed by either or both of two gas chlorinators each having a capacity of 400 lb. per 24 hr. Facilities are provided for the storage and handling of ton chlorine containers. Control of the cyanide treatment process is manual, based on maintaining a predetermined free chlorine residual in the tank effluent.

From the acid holding tank, the acid-chromium wastes go by gravity through an automatic rate controller and flow meter to the tank in which the hexavalent chomium is reduced to the trivalent form. This tank has a volume of 6,000 gal., providing a detention period of 60 min. at the design flow of 100 g.p.m. A mechanical mixer installed in this tank provides about 69 turnovers per hour. Sodium metabisulfite is added in the inlet line to the tank. The amount of bisulfite fed is automatically controlled, based on the oxidation-reduction potential of the tank contents.

The treated alkaline and acid wastes are brought together in a tank where the free acid is neutralized by the addition of hydrated lime. This tank provides a detention of about 10 min. at a total flow of 200 g.p.m. A mechanical mixer provides about 141 turnovers per hour. The amount of lime added is manually controlled.

Metals Removal

From this tank, the neutralized combined wastes flow to a combination flocculation and settling tank, known commercially as a Cyclator. In this tank, the addition of lime is automatically controlled to maintain a uniform pH as required. All of the metals are precipitated as hydroxides. The clarified liquor is withdrawn over a circumferential weir, and flows by gravity to the city's sewage treatment plant. The settled sludge is withdrawn automatically at regular intervals to a sump, from which it is pumped to lagoons for further concentration and ultimate disposal. No skimming device is provided in the Cyclator because the small quantities of oils present can be entrained by the hydroxide floc. The treatment tanks are shown in Figure 3.

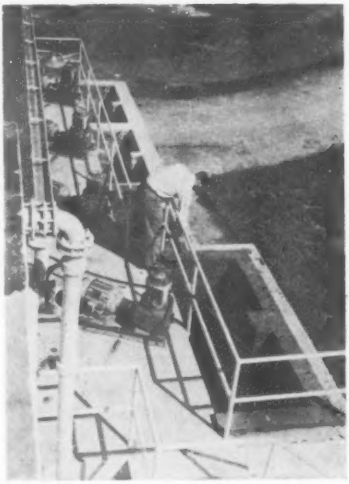

FIGURE 3.—Chromium, mix, and cyanide treatment tanks, where waste is brought to desired stage prior to flocculation and settling.

Chemical Feeds

The lime and sodium metabisulfite are fed from dry-type chemical feeders. Because these feeders were located in the basement of the control building, it was necessary to pump the solutions to the point of application. The feeders are designed to maintain a constant per cent chemical concentration in the dissolving chamber of the feeder. From each feeder the slurry is pumped to a separate splitter box. Each box is provided with a rectangular weir, over which the entire flow passes. A splitter plate is provided to split the

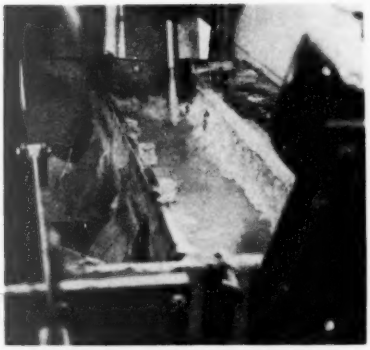

FIGURE 4.—Splitter box for proportioning flow of lime slurry to mix tank, flocculator, and slurry return.

flow over the weir, so that the desired portion goes to the point of treatment and the excess returns to the feeder dissolving chamber. Where automatic control is required, the position of the plate is set by a diaphragm-type air operator.

Figure 4 shows the splitter box used for the lime. Two weirs, each with splitter plates, are provided. One controls the lime fed to the mix tank and is manually controlled. The other controls the lime fed to the Cyclator, and is automatically controlled.

The use of this type of splitter box is more satisfactory than throttling valves in the pump discharge line, particularly in the case of lime where restricted openings result in plugging. Also, there is practically no limitation to the slurry concentrations that can be used, nor on the quantity of slurry pumped. An unusual amount of flexibility is provided, in contrast to other control methods.

For the automatic control of the sodium metabisulfite fed for chromium reduction, a platinum-calomel electrode pair is provided in the treatment tank to measure the oxidation-reduction potential of the waste. This potential is received by a remote indicator-recorder-controller instrument, which in turn pneumatically positions the air operator on the splitter box. This function is entirely automatic and will maintain any predetermined potential in the tank contents. By maintaining a sufficiently low potential, the reduction of the hexavalent chromium is effected. A typical curve showing the relationship between potential, hexavalent chromium, and quantity of reducing agent added is shown in Figure 5. Kelch and Graham (3) have presented a complete explanation of this method

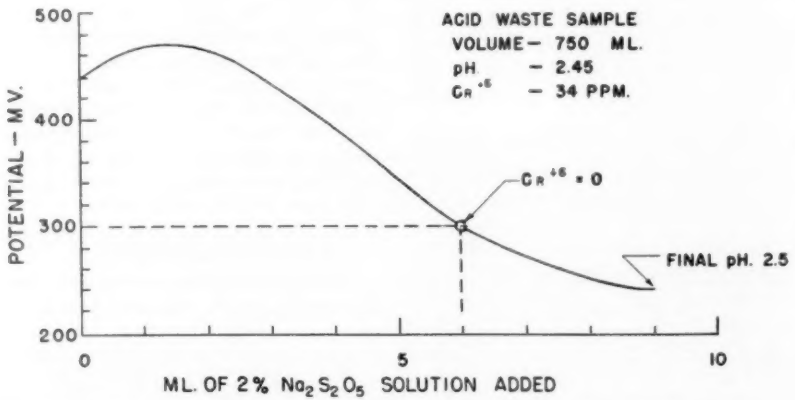

FIGURE 5.—Relationship between oxidation-reduction potential, reductant, and hexavalent chromium.

of control. Although they used ferrous sulfate instead of sodium metabisulfite as the reducing agent, the theory and application are comparable.

For the automatic control of the lime fed to maintain a constant pH in the mixing compartment of the Cyclator, the same principle is employed as for the oxidation-reduction control. The only difference is that a glass electrode-calomel electrode pair, which measures pH instead of O-R potential, is used.

A spare chemical feeder was installed. It was arranged so that it could also be used to feed a coagulant to the mixed wastes entering the Cyclator if required.

Other instrumentation is provided in addition to that already described. Rate of flow totalizer-indicator-recorder instruments are provided for both the raw acid and alkaline wastes. "Bubble-tube" type water level indicators are provided for each of the raw waste holding tanks. All instrumentation is remote and operated pneumatically.

Piping and Tank Linings

All pipe carrying the acid wastes is Duriron. Type 316 stainless steel is used for all equipment, such as pumps and mixers, coming in contact with the acid waste. Rubber-lined valves are used on acid waste lines. Cast iron or steel is used for all pipe, valves, and equipment coming in contact with the alkaline wastes.

The acid waste storage tank, the chromium reduction tank, and the mixing tank were originally lined with a four-coat system of a furane derivative base acid-resistant material. The lining failed during the first two weeks of plant operation, so this material was removed and the tanks were lined with acid-resistant brick. The brick lining is 8 in. thick in the acid waste holding tank and 4 in. thick in the chromium reduction and mixing tanks.

Sludge Disposal

Ultimate disposal of the sludge from the Cyclator is to three lagoons having a total area of about 0.75 acres. Each lagoon is provided with a swivel type of overflow pipe so that clear supernatant and excess storm water can be withdrawn down to any desired level.

Control Building and Laboratory

A control building is provided for housing all of the equipment. The laboratory, chemical storage area, chlorinator room, and chlorine storage area are located on the ground floor. The meter panels and the electrical motor control center are in the laboratory. Chemical feeders can be charged from this floor. The chlorine cylinder storage area is in an attached semi-protected part of the building. The only entrance to the chlorinator room is from this "outdoor" area. Chemical feeders, pumps, and miscellaneous equipment are located in the basement. All of the main piping and valves are located within the building to facilitate repair and maintenance.

Provisions were made so that up to 15 g.p.m. of the final effluent could be re-used in the treatment plant. At present, this is a water source for meter flushing connections, the chemical feeders, and the chlorinators. For the present two-shift operation of the plant, this means a saving of about 20,000 g.p.d. of water. This re-use of water, together with the reduction of water used in regular plating operations as recommended in the survey report, has resulted in a saving of about $5,000 per year in purchased water.

Plant Operation

The treatment plant was completed and ready to put into operation late in February, 1951. The first three weeks were required for field adjustment of the equipment and calibration of instruments and controls. During this period, wastes were treated only 8 hr.

TABLE II.—Summary of Plant Operating Data [1]

Sample	Flow (g.p.d.)	pH	CN (p.p.m.)	Cr (p.p.m.)		Cu (p.p.m.)
				Total	Cr^{+6}	
Alkaline waste	16,110	11.4	22	—	—	—
Acid waste	79,510	2.0	—	51	38	—
Final effluent	95,620	8.8	0.1	3.1	1.9	1.1

[1] Average daily values.

per day. Since then, the plant has been operated 16 hr. per day to coincide with the plating room schedule. A complete record of analyses is not available for each day of operation. A summary of the available data is shown in Table II. Corresponding data on chemicals used are given in Table III.

Operation of the plant has not yet reached the routine stage. Refinements of operational details still remain to be worked out. For this reason, the data do not indicate ideal results. For example, the quantities of chemicals used for the treatment of the cyanides and hexavalent chromium are high by comparison with experience reported for some other plants. The quality of the effluent, as based on analyses of composite samples, shows that it is very close to the requirements established by the city of Delaware. Quantitative analyses for oil have not been made, but visual observation of the final effluent shows only traces of oil. The oils in the raw wastes are being effectively removed with the floc in the Cyclator.

It has been found that the metallic hydroxide floc formed settles readily. Use of a coagulant in the Cyclator has not been required. The sludge as withdrawn settles to about 75 per cent by volume in 30 min.

Figure 6 shows samples of the raw wastes and final effluent.

The experience with the sodium metabisulfite has not been entirely satisfactory. After periods of shutdown, the chemical cakes in the bottom part of the feeder hopper. This results from absorbing moisture from the saturated air within the feeder and above the water surface in the dissolving chamber. Excessive amounts of SO_2 gas are frequently liberated, both in the dissolving chamber of the feeder and in the splitter box, creating an undesirable condition within the control building. This has been eliminated by installing a blower to exhaust the gas from both places to the outside. Also, severe corrosion has been observed at several points inside the feeder. None has been observed, however, in the storage hopper where no moisture is present.

TABLE III.—Chemicals Used[1] under Operating Conditions of Table II

Chemical	Amount Used (lb.)
Lime, $Ca(OH)_2$	510
Sodium metabisulfite, $Na_2S_2O_5$	144
Chlorine	56

[1] Average daily values.

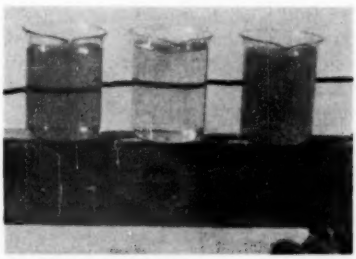

FIGURE 6.—Samples of raw chromium-acid waste (left), final effluent (center), and raw cyanide-alkaline waste.

Maintenance Schedule

The first months of operation of the plant have indicated the likely sources of difficulty. A regular maintenance schedule has been set up to prevent interruption of operations by remedying the source of the trouble. The oxidation-reduction electrodes and the pH electrodes must be cleaned at least twice a week. Both sets of electrodes become coated with a thin film of oily material, which is present in the wastes.

Soot from railroad locomotives passing the plant has been a constant source of trouble. The soot, together with particles of vegetation that settle on the surface of the Cyclator, is picked up in the treated effluent, which is recycled to the chemical feeders and chlorinators. Because of this, it is necessary to clean the strainers on the inlet water lines to the chlorinators once a week. Also, the rotameters and solenoid valves on the water lines to the chemical feeders must be flushed and cleaned at least once a week.

Normal preventive maintenance on other pieces of equipment has proved to be adequate.

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Stream Pollution

AN INTEGRATING WATER SAMPLER

By Harold A. Thomas, Jr., R. Stevens Kleinschmidt, and Frank L. Parker

Associate Professor of Sanitary Engineering, and Graduate Students, respectively, Department of Engineering, Harvard University, Cambridge, Mass.

A reliable, rugged, and inexpensive device for collecting integrated water or sewage samples has been developed in connection with a research program sponsored by the United States Atomic Energy Commission. In this project the effectiveness of self-purification mechanisms of natural bodies of water in reducing the concentration of radioactive compounds is being assessed.

The test procedure has been to dose an isolated reservoir with varying amounts of different short half-life radioisotopes. The dispersion and disappearance of these caused by mixing, dilution, precipitation and deposition, absorption, and biological uptake have been traced by measuring the radioactivity in samples collected at various distances from the point of dosage.

A "slug" of radiocontaminant introduced at a point in the water tends to disperse under the stimulus of the wind and other processes that induce mixing. Such dispersion is apt to proceed erratically, since the mixing action is seldom isotropic and steady. The "tracer cloud" formed is usually irregular in outline and density. The original mass often breaks up into smaller masses that may move in different directions. It is manifest under these circumstances that composited or integrated samples are needed to provide an accurate indication of conditions at a given station. Here grab samples are practically useless, as is the case in many other sampling situations.

The principle of the sampler may be described with reference to Figures 1 and 2. The apparatus consists of a vacuum can (which also serves as a float), a needle valve, and a sample collection bottle. In operation the can is evacuated with a modified bicycle pump. The suction thus created is relieved by a slow air leak through the needle valve causing water to be drawn through the inlet tube into the bottle. The rate of intake is set by connecting a graduated pipette to the needle valve and immersing the lower end of the pipette in a beaker of water. The needle is then adjusted to give the proper rate of flow—for example, 0.7 ml. per min. if it is desired to collect one liter during a 24-hr. period. The pipette is then removed and the sample bottle put in place. Finally, the sampler is anchored at the desired location and depth.

In placing the sampler below the water surface, precaution is taken to prevent hydrostatic pressure from causing an unwanted initial inrush of water. To do this, a pressure of air equal to or greater than the hydrostatic pressure is put in the sample bottle with the pump just before it is set down, the inlet tube being shut off with a remote-release pinch-cock. When the apparatus is in position, the pinch-cock is released and retrieved by a string. The excess air immediately bubbles out, equalizing the pressure, and the sampler starts operating.

The critical part of the assembly is the needle valve. Several types of these

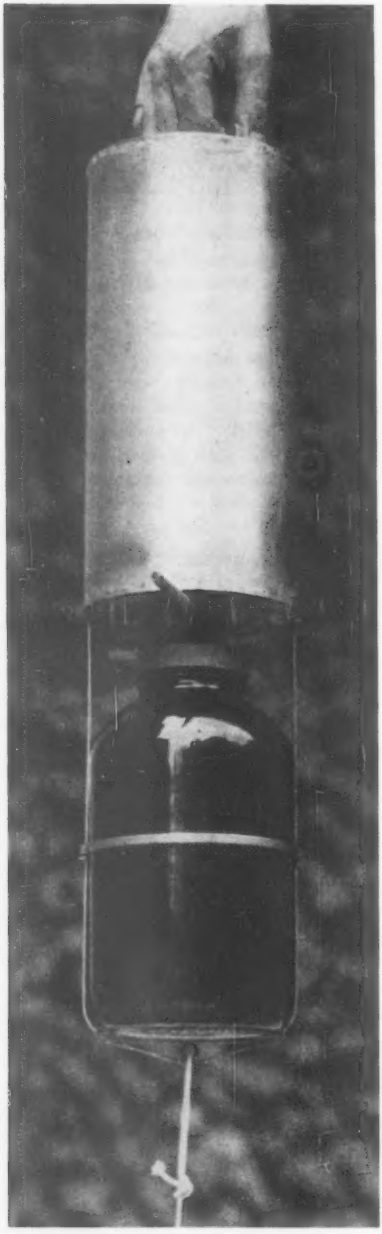

FIGURE 1.—Construction of integrating water sampler.

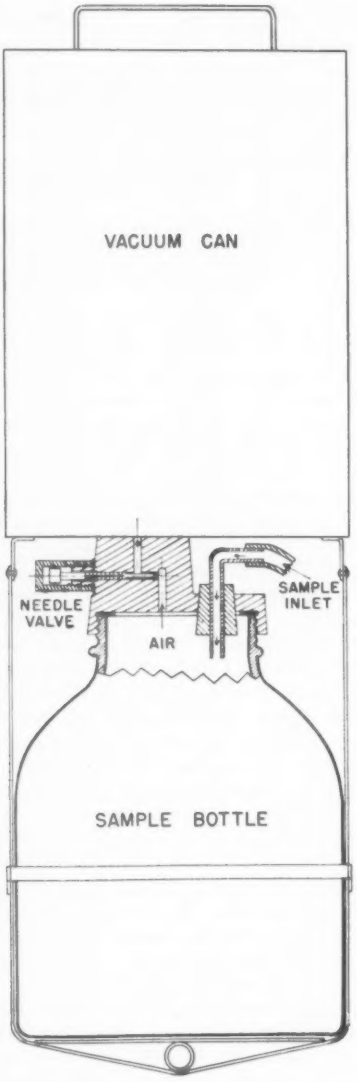

FIGURE 2.—Design of integrating water sampler. Vacuum in can at top creates slow air leak through needle valve, causing water to be drawn through inlet tube into sample bottle. The sampler, when anchored at desired location and depth, will collect an integrated sample for any predetermined length of time between 10 min. and 36 hr.

were tested. The final design, evolved by Mr. Kleinschmidt, is highly satisfactory; with it air-leak rates may be set so as to give collection times ranging from a few minutes to 36 hr. The size of sample depends upon the capacity of the vacuum can. In the sampler shown in Figure 1 the vacuum chamber has a volume of 5,560 ml.; this is enough to provide a pressure gradient of sufficient steadiness to collect a 2-l. sample in 24 hr. at a nearly constant rate. The over-all height of the apparatus is 24 in.

If the sampler is inadvertently left in the water for a period longer than the collection time set, the sampler will automatically stop collecting, as the constriction at the needle valve is so great that it will not pass water at an appreciable rate.

The inlet tube may be of any diameter or length. The device may be used to sample raw sewage; it may be operated in rapidly moving streams or in reservoirs subject to wave action.

The authors will be pleased to furnish to interested persons additional information relating to design and construction.

COMMUNITY RELATIONS IN AIR POLLUTION ABATEMENT

Hostility to the industrialist on the score of air pollution, indeed anti-pollution laws themselves, are results of failures in community relations, according to the "Community Relations" chapter of the "Air Pollution Abatement Manual," which is being published chapter-by-chapter by the Manufacturing Chemists' Association, Inc.

Prepared to assist members of the chemical industry in achieving abatement of air pollution by the most effective means, and at the lowest ultimate cost, the Manual includes the best technical information available on the handling of pollution problems. It also suggests how industry can work with its employees, its neighbors, and public officials to achieve a spirit of understanding, cooperation, and mutual good-will.

A comprehensive program for the individual company is outlined in considerable detail by R. J. Bulkley, who wrote the Community Relations chapter, which the M.C.A. has recently published as "Manual Sheet P–4." Simultaneously, it has published "Manual Sheet P–2," in which J. M. Gillet presents an introduction to the Manual, discussing such phases of the problem as its historical background and recent developments, as well as general and specific responsibility for abatement of air pollution.

These are two of the Manual's 13 chapters, which will be published separately as completed over the next 12 months. Subsequent subjects will include "Legislative Requirements," "Physiological Effects," "Sampling Procedures and Measuring Equipment," "Analytical Methods," "Meteorology of Air Pollution," "Dust and Mist Collection," "Gas and Vapor Abatement," and "Evaluation of Data."

Sponsored by the Air Pollution Abatement Committee of the M.C.A., each chapter of the Manual is available, on publication, from the Association's Washington office (246 Woodward Building, Washington 5, D. C.) at 25¢ per copy.

THE OPERATOR'S CORNER

CONDUCTED BY HERBERT P. ORLAND

". . . ALL THE POTENTIALS OF TIME BOMBS"

Any sewage treatment plant operator who has the slightest inclination to allow familiarity with hazardous ventilation conditions to lull him into an it-can't-happen-here attitude certainly should read with extreme care the paper on "Ventilation of Sewage Plants," presented at the 1951 annual meeting of the Michigan Sewage and Industrial Wastes Association and published elsewhere in this "Corner." Here is the observation and discussion of an expert on ventilation problems, brought to bear directly on conditions obtaining in the daily work of the operator.

How strongly the author, Mr. Robinson, feels about the matter can be seen from his remarks prefacing presentation of the paper, as follows:

"It gives me a good deal of pleasure to appear before you gentlemen. Not that I am so anxious to speak, but rather that I feel very much honored to be allowed a few minutes in your presence. The reason I say this is that I sincerely believe you to be the most righteous group in the country, or you would undoubtedly all have been blown to bits before this.

"Some weeks ago I spent two days with the secretary of the Michigan Sewage and Industrial Wastes Association touring some of the treatment plants in the state. Upon returning home I picked up the local paper and noticed that one of the local theaters was advertising a double feature of horror movies because it was Friday the 13th. This prompted me to tell my wife that I had just had a taste of such double features, inasmuch as I had had two days in plants that contained all the potentials of time bombs."

Generally the operator has had no part in designing his plant and its ventilation facilities. However, he has nobody to blame but himself if inadequacies found later are not remedied. With the advanced state of present ventilation know-how and equipment, no such hazardous conditions as mentioned by Mr. Robinson should be allowed to exist.

Just opening vents and placing exhaust fans willy-nilly in potentially hazardous areas is not the solution. Serious consideration of all the factors is necessary. The starting point, of course, is a complete survey of the plant to locate the ventilation hazards. Then each potentially dangerous condition should be analyzed carefully as to its peculiar requirements and steps should be taken to remedy it on that basis.

Don't continue to sit on the time bomb and hope it won't go off. Unarm it and get rid of it!

H. P. O.

VENTILATION OF SEWAGE PLANTS *

By K. E. Robinson

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Without intending to be an alarmist, a few of the hazardous conditions observed on a recent two-day tour of some of the sewage treatment plants in Michigan may be of interest. All of the conditions mentioned result from faulty ventilation or lack of ventilation—all are correctible by proper application of the principles of good ventilation.

One of the first installations visited was a screening room displaying an open gas burner within a few feet of the open sewer. As this was a confined space, dire consequences could result if gasoline, naptha, or other flammable liquids were accidentally dumped into the sewer. At another plant, where an explosion had occurred some time previously, a fan had been installed to ventilate the area. However, there was no assurance that the air movement would take place where the maximum concentration of flammable materials would originate. It might be added that explosion-proof fixtures had been installed in the area, but an extension cord was being used with the fixture removed.

There was another case of faulty controls on a boiler using sludge gas allowing the gas valve to be opened when the pilot light was off. One or two men had been singed while trying to light the boiler under these conditions. There were several instances of men working in tunnels where the greatest hazard of fire or explosion was between them and the door. An accident would have literally "sealed their fate." One plant had gas leaking from the water trap on the gas line, with the possibility of high concentrations being developed.

It seemed to be common practice for all men to go into pumping stations, even of the underground type, without any means of determining whether there was an explosive mixture present, or even whether there was enough oxygen to support life. The Federation has published a manual warning against these hazards and giving some suggestions as to their elimination. Nevertheless, further discussion of the control of some of the hazards found in sewage treatment plants seems in order.

Explosive Range

There is a feeling that any area can be made safe if any type of ventilator is placed in the ceiling. This feeling apparently stems from the fact that methane or marsh gas is "lighter than air." The density of methane is 0.55, compared to 1, which is standard air. It must be remembered, however, that the value of 0.55, which shows that the weight of methane is about one-half that of air, holds true only for pure methane. In speaking of those concentrations which will burn, the explosive range must be understood and considered. The explosive range includes all concentrations of a mixture of flammable vapor or gas in air in which a flash will occur or a flame will travel if the mixture is ignited. The lower explosive limit is the lowest percentage at which this occurs and the upper explosive limit is the highest percentage.

Needless to say, a mixture can be within the lower explosive limit and contain comparatively little methane in the mixture. The lower explosive limit of methane is a 5 per cent mix-

* Presented at 1951 Annual Meeting, Michigan Sewage and Industrial Wastes Assn.; Grand Rapids, Mich.; June 4–6, 1951.

ture, or 50,000 parts per million parts of air. Calculation on this basis shows that the lower explosive limit has a vapor density of 0.9775. If it is further considered that safe practice dictates a maximum concentration of 25 per cent of the lower explosive limit, vapor density of 0.994375 should not be exceeded. As can be seen, this varies considerably from the vapor density of the pure gas and should be taken into consideration in designing ventilation.

Perhaps the biggest hazard other than methane is the danger of fire or explosion when gasoline, lacquer thinners, solvents, etc., are spilled into the sewers. Apparently there has been a feeling that if this happens all of the vapors would be heavier than air and ventilation would be required from the floor. Although gasoline vapors are 3.5 times as heavy as air, the vapor density of the mixture at the lower explosive limit will be only 1.0325. Once again, if the concentration is kept to 25 per cent of the lower explosive limit, the vapor density is only 1.008125.

Ventilation Location

The main purpose in bringing attention to the vapor density of these two mixtures is to impress the fact that if proper ventilation is employed and mixtures are kept below the lower explosive limit, it is immaterial where the air is exhausted. The important thing is the location of the air supply in relation to the point of exhaust. This is true with any ventilation problem, but even more so in this type of work, due to the airtight construction of most of the rooms and passages.

Some problems which are typical of those found in pumping stations and disposal plants will illustrate the point. Figure 1 shows a sketch of a typical digestion control building. It was copied from the actual construction plans, although only a little of the piping is included. This particular room is

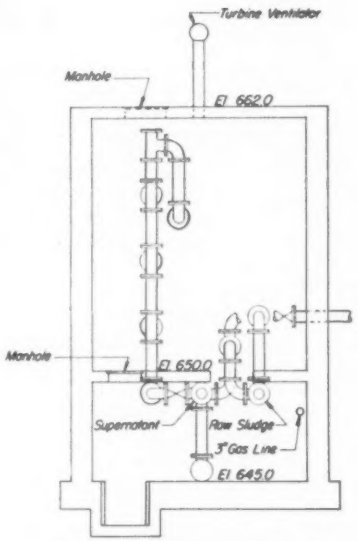

FIGURE 1.—Typical digestion control building construction plan, showing grossly inadequate ventilation provisions.

constructed below ground level; the walls, ceiling, and floor are made of reinforced concrete. The only entrance is through the manhole at the top, with a secondary manhole for access to the bottom chamber. The only ventilation shown on the plans is a 6-in. pipe topped by a turbine ventilator. Inasmuch as there is no reason to expect a temperature differential and it has already been shown that the vapor density of a mixed gas is nearly that of air, the gravity ventilator could not be expected to be of much value unless there was a steady wind. If it is assumed that such a wind is to be had at all times, it is still impossible to see how any air motion can be expected through a chamber made of watertight concrete except for one small opening, which is sealed with a cast-iron plate. Even if the cover were left off there would be no reason to believe that air would find its way to the bottom of the chamber and then to the ventilator. With the

arrangement shown it is not believed that it would ever be safe for a man to descend into the chamber without first making a complete check of the atmosphere with instruments.

It might be of interest to know how much gas would be required to create an explosive mixture in this building, which is 8 ft. by 10 ft. by 17 ft. The volume, disregarding the space taken up by piping, is 1,360 cu. ft.; if 68 cu. ft. of gas leaked into this area, an explosive mixture would result.

Another typical example is shown in Figure 2. In this case, the office is above ground, with the typical equipment rooms, including pumps and motors, beneath. Off the second level is a tunnel running some 50 ft. to a digester control building, which is partially below ground with a small superstructure above the grade. The ventilation specified included a two-speed fan exhausting from the bottom level of the main building at the rate of 410 cu. ft. per min. at high speed and 230 cu. ft. per min. at low speed. There was also a unit specified capable of handling 460 cu. ft. per min. and exhausting from the bottom level of the digester control building. If both fans were in continuous operation they should exhaust 870 cu. ft. per min. However, the area which they are supposed to control includes about 20,000 cu. ft. and this would mean that they would provide about 2.5 air changes per hour. Besides this 870 cu. ft. of air required by the fans, air will be required for combustion by the sludge heater. Of the total volume of the building, only a small portion is above ground and there is little possibility for infiltration. Experience has shown that under such conditions there is extreme danger of the occupants of the building being exposed to carbon monoxide, because the fans cause a downdraft through the heater vent pipe.

The results of inadequate ventilation are well illustrated by Figures 3 and 4, which show a plant after an explosion in the tunnel near the digester. The operator had been in this area only minutes before and had just returned to his office. In spite of the wreckage shown, he was lucky enough to escape serious injury.

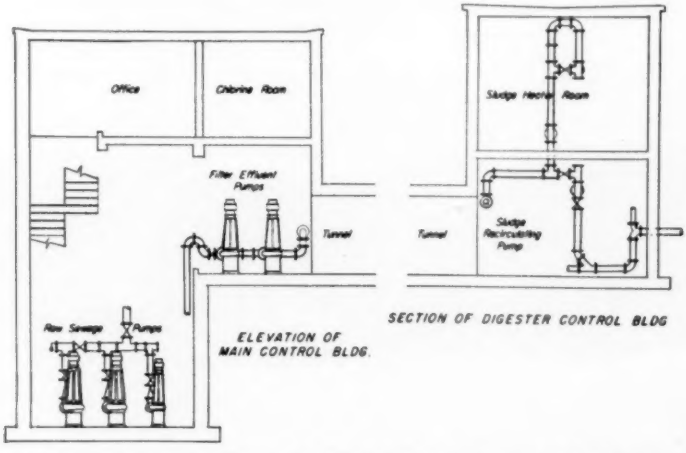

FIGURE 2.—Typical layout of control building and digestion control building connected by tunnel.

Ventilation Control Methods

Other examples could be discussed, but those already mentioned are typical of the problems found in this type of plant. The pumping stations can very easily be treated the same as the digester control buildings. Therefore, consideration is directed to ventilation control procedures.

Referring to Figure 1, it is self-evident what is needed. It has already been shown that the gravity system could not be expected to work and that there was no way for air to get into the building. Therefore, it would seem that a reasonable solution would be to arrange an air supply at the top of the building and exhaust air at the floor level of the lower chamber by means of a mechanical system. Inasmuch as the potential hazard is present at all times, the fan should operate continuously.

FIGURE 3.—Front view of control building damaged by explosion in tunnel near digester.

FIGURE 4.—Rear view of control building damaged by explosion in tunnel near digester.

The second example discussed (Figure 2) is somewhat more complicated because of the different equipment and the larger area involved. Nevertheless, the basic principle is still the same and for this plant it is suggested that specifications include a tempered air supply unit to furnish proper volumes of air to the office with mechanical systems to exhaust air from the lowest point of the main building and the upper level of the digester control building. These exhaust systems will draw the supplied tempered air through both the buildings and the tunnel and should keep the entire area well ventilated. It should be remembered, of course, that the air supply unit will have to supply air for both the exhaust fans and for the heaters.

The discussion thus far has made no mention of ventilation in the chlorine room. The original plan, from which Figure 2 was copied, showed a typical kitchen exhaust fan at the floor level. This would undoubtedly be satisfactory, except that there is only one opening to the room and when this is closed there would be no air inlet. Here again it is strongly suggested that arrangements be made so that automatic louvers on an air inlet are opened whenever the exhaust fan is in operation.

How Much Air

No figures have been given regarding air volumes. There has been very little work done to determine the proper volumes of air for satisfactory control. However, such a study was made by the Bureau of Industrial Hygiene and the Division of Industrial Safety in the State of California. The report specifies:

"If continuous ventilation is used at least 10 air changes per hour should be provided for dry wells and 25 air changes per hour for wet wells. If intermittent ventilation is employed, the system should be turned on at least 15 minutes before anyone enters the pit and should be operated at a rate of 60–70 air changes per hour. . . . Intermittent ventilation should be avoided . . . in favor of continuous ventilation."

Other recommendations include the statement:

"All pumping pits, especially if over 6 feet deep, should be provided with adequate ventilation. The exhaust duct location for the pits should be located so that contaminated air is removed near the bottom of the pit.

"Safety equipment, including approved breathing apparatus and a life line and harness, and test equipment, including a flame safety lamp, a hydrogen sulfide detector and a small combustible gas indicator, should be provided for each manhole, pumping pit or other underground crew. Each crew should be composed of at least two workers. No employee should be required or permitted to enter a manhole unless a watcher remains at the surface."

Conclusion

The author has tried assiduously to avoid giving the impression of being an alarmist. In fact, he has purposely been very conservative in his statements, and avoided discussion of the dangers of hydrogen sulfide, oxygen deficiencies, and other hazards of the profession. His purpose will have been accomplished, however, if he has aroused some awareness of the hazards attendant on lack of adequate well-designed ventilation in the potential danger spots of sewage treatment plants.

SELECTION OF CORROSION-RESISTANT MATERIALS FOR WASTE TREATMENT PLANTS *

By Walter J. Sandel

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Waste treatment plants may be roughly divided into two types. The first comprises municipal sewage treatment plants, which normally deal with corrosion problems wherein the attacking materials are water, oxygen, such additives as ferric chloride or ferrous sulfate in an acid solution, and hydrogen sulfide, which at times oxidizes to sulfuric acid. The second type comprises industrial waste treatment plants, which are called upon to handle many more and different corrosive materials. Both types of plant must treat the waste material to such a point that it is acceptable in streams and rivers without polluting them. The industrial waste treatment plant may discharge its effluent into the sewers of the municipality; when it does, it will be required to meet the standards set up by the municipality relative to the character of the effluent.

Water, Abrasion, and Chemical Reaction

From the foregoing it is seen that both types of plant are called upon to process wastes that are corrosive, and often abrasive, to the materials used in the construction of the treating equipment. It is well to note that buildings may also be attacked, due to the evolution of fumes, which, either by themselves or in combination with water, will react with the structural member. Water is a concomitant of waste treatment. It is used as the transporting medium for the wastes, as a diluent, and as a cleaner. As a result, there is the continuous problem of separating the water from the equipment. As the water flows through the treatment plant it is called upon, in many cases, to carry solids as well as solutions. The solids abrade the surfaces with which they come in contact, thus wearing down the thickness of any protective skin and allowing moisture to penetrate through the protective material. This moisture, plus dissolved oxygen and other chemicals, will react with the base materials of construction to change them and, usually, destroy them. The protective skin may be either softened or dissolved by the action of some solvent which is part of the industrial waste being treated, thus baring the base material of construction to attack. Thus, the problem becomes one of either building with materials which are in themselves chemically inactive, insoluble, and abrasive resistant to the waste being treated, or protecting them with an applied skin having these properties plus a high resistance to moisture penetration or transmission. As the base materials of construction are often chosen for reasons other than their corrosion resistance, it behooves the operator to choose wisely and well the protective skin which he will apply over the equipment and plant structural materials.

Factors Affecting Selection of the Corrosion Resistant Skin

To make an intelligent choice of protective skin, several factors must be taken into consideration. In the order of their recognition they are:

1. The properties of the waste products.

2. The requirements of the state and local authorities regarding the acceptable effluent.

* Presented at 1951 Annual Meeting, Ohio Sewage and Industrial Waste Treatment Conference; Columbus, Ohio; June 20–21, 1951.

3. The properties of the available materials of construction.

4. The properties of the available materials of protection and the availability of qualified and proper application of them.

5. The economics of the project.

Properties of the Waste Products

The properties of the waste products requiring treatment must be known before any choice can be made regarding either the materials of construction or the materials of protection. The questions to be answered include:

1. What will be the temperature at which the wastes will be treated? It is important to know not only the incoming temperature of the waste, but also the temperatures which may result from treatment of it. A protective skin which will do an excellent job at 70° F. may fail miserably at 150° F., or it may have properties which make it undesirable at 0° F.

2. What is being treated? Is it acid, alkali, oil, salt, solvent, in solution or suspension, or is it abrasive? Will it be changed into anything that will require a different protective skin than the one used at the point of influent?

3. At what concentration will the waste be treated? What will the concentration be throughout the entire processing of the wastes? Are there likely to be "hot spots" with reference to concentration of the mixture of waste material and treating chemical? If so, an endeavor should be made to keep this hot spot from coming into close proximity with the protective skin.

4. Will there be splash and/or fumes developed during the treatment processes? Will the processing require any kind of treatment involving the possibility of mechanical damage?

All of the above must be taken into consideration, for they all have a bearing upon the type of protective skin that may be safely used. In any event, the protective skin, in most cases, must be quite versatile in its ability to cope with a variety of solutions.

State and Local Laws Regarding Effluents

State and local laws regarding acceptable waste effluents vary from point to point, but not to any large degree. This variation is natural, as there are many factors affecting stream conditions. However, there seems to be general acceptance of the theory that all industries should shoot at a pH of 7, with anything lower than 5.5 or higher than 9.0 being unacceptable. The important thing to recognize here is that almost all wastes require treatment, and that most wastes are corrosive. There are sludge types of waste which are capable of corrosive action and which pose the possible problem of mechanical damage in the form of abrasion or due to carelessness on the part of a workman who has been given the task of removing sludge from a treatment basin. State and local laws generally bar any type of effluent which can cause damage to existing sewer facilities or disposal equipment; or which is polluting in its effect on streams and rivers. The extent of treatment required for making the industrial waste acceptable must be known.

Properties of Construction Materials

Speaking generally, the most common materials of construction are steel, masonry, and wood, which have many and varied properties. Their resistance to corrosion is generally very poor. Fortunately, all of them will take and hold a protective skin. The method of surface preparation is not the same for all of these materials; and if there are included some building specialties, such as corrugated asbestos, galvanized iron, etc., each material must be treated differently prior to application of the protective

material. Masonry and concrete structures have a habit of cracking due to settlement and/or shrinkage. A small amount of this is overcome and does no harm, providing the proper covering is selected for the job. Certain standard steel structural members are fabricated with lapped areas, which prevent proper surface preparation and covering; they should be sealed with a weld prior to surface preparation.

Surface preparation for the common building materials has been somewhat standardized; for top quality and lasting service such preparation consists of sandblasting for steel, acid etching for concrete and masonry, and sanding for wood. There are other methods of surface preparation which are practiced, such as wire brushing, scraping, solvent washing, etc., but the life of the protective coating will be directly in proportion to the quality of the preparation. The best surface preparation possible should be used, as it will add life to the poorest coating.

From the foregoing it can be seen that it is necessary to know about the materials of construction contained in the plant and its equipment. Also essential is a knowledge of the methods of surface preparation. These, plus the nature of the waste product, have a bearing upon the choice of protective material.

Properties of Available Protection Materials

Before the advantages and disadvantages of any protective material can be intelligently weighed, one should know about and have complete data on its properties and application procedure. With reference to its properties, the person making the choice will want to know about its composition, coverage, drying requirements, finish, maintenance, flexibility, adhesion, abrasion resistance, electrical resistance, temperature resistance, combustibility, thermo-conductivity, water absorption, moisture vapor transmission rate, film thickness, weather resistance, acid resistance, alkali resistance, solvent resistance, and salt resistance. Also, its resistance to mineral, vegetable, and animal oils; its toxicity; and its resistance to any special condition involved in the particular problem at hand.

Composition

There are many makers of protective coating and covering materials, just as there are many makers of anything else one buys and uses. All stainless steel is called just that, but as is well known, there is a difference; it is this difference which makes one type of stainless steel work well under certain conditions, whereas another fails under the same conditions. Composition, then, is a factor in the ultimate success or failure of any material formulated to resist corrosion. The choice of coating material, therefore, will be limited to those makers whose formulations have proven themselves.

Coverage

Coverage is an elusive thing, and in protective coating work is usually stated: "Not to exceed (certain specified areas) per gallon per coat." This is done to attain a certain minimum thickness per coat, with multiple coats being applied to achieve the proper final thickness. The coverage per gallon per coat will vary as the smoothness of the surface to be covered varies. A smooth dense surface will not require as much material as a rough porous one. The specified coverage should not be exceeded at any time.

Drying

Drying requirements may vary from as little as 5 min. to as long as a week. These requirements must be taken into consideration when allotting total time for the application of the selected coating material. The requirements may

call for baking or forced drying, either of which require equipment and know-how that can be obtained from any qualified applicator of protective coatings and coverings. More on the subject of application is given later on in this discussion.

Finish

The finish of the completed coating or covering may or may not be important to the requirements of the job. In most cases both good appearance and excellent resistance can be had. In fact, there are many installations where the protective coating is the decorative coating. Many color schemes are possible with protective coatings.

Maintenance

The primary interest may be in the requirements of the protective material relative to maintenance operations. This calls for selection of a material that can be repeatedly scrubbed with the usual cleaners found in industry without detrimental effect, and that can be repaired by simply cleaning the surface thoroughly and then re-coating. In the case of a sheet application, the selected material should be repairable at the job site on short notice.

Flexibility

Frequently it is desired to apply a protective coating to a surface that is both flexible and subject to accidental flexing or bumping. This calls for a coating or covering that is inherently flexible; one that will go and come with the base material, whether it be due to expansion and contraction from temperature variations or other means.

Adhesion

Adhesion of the applied coating is a very important factor in the life of the coating. In fact, once a protective material has been selected that is chemically inert to the waste materials, every operation is directed toward achieving the best possible adhesion. Adhesion varies with the type of material selected; but here again the quality of application and the type of surface preparation have a definite bearing upon the adhesion of the protective coating or covering. A material should be selected which is capable of good adhesion in the hands of a qualified applicator.

Abrasion Resistance

Obviously, it is desirable that the material will have very good abrasion resistance. This type of resistance is not necessarily a property of all types of coating materials. Because abrasion may vary from soft rubbing to harsh gouging or scraping, the protective material should be selected to meet the type of abrasion inherent in the waste treatment procedure. Many times it is found that the protective material having the necessary chemical resistance needs to be fortified with a ceramic material to protect it from harsh abrasion. This is especially true in waste treatment operations that embrace periodic removal of sludge by mechanical means, such as shoveling, raking, or scraping.

Electrical Resistance

Electrical resistance of the protective material may play a part in the problem of waste treatment. If the selected material has high di-electric strength, it will effectively control, and in many cases prevent, electrolytic corrosion. As this type of corrosion works very fast, it is important to choose a protective material having a high di-electric strength whenever this type of problem is present.

Temperature Resistance

There are many good protective coating and covering materials which will work excellently at temperatures between 50° and 100° F. As the temperature goes up, their effectiveness goes down; and as the temperature goes

down, their effectiveness goes up, except as to embrittlement and adhesion. This is because most coating materials, when subjected to increased temperatures, soften, become more permeable, and will more readily pass moisture, vapor, and gases. They also become more readily dissolved in all solvents. As the temperature goes down, most coatings will become more dense, and if they retain adhesion will resist the passage of moisture and gases more effectively. They also crack and chip more easily. For this reason it is highly desirable that the waste treatment be operated at medium temperatures and protective materials be selected that have a wide range of effectiveness with changes in temperature.

Noninflammibility

It is frequently necessary to apply a protective coating to the walls and ceilings of the waste treatment plant. One that is noninflammable after application is highly desirable, as it will reduce the hazard of fire and at the same time reduce the insurance rate. It is always good practice to select a coating which will not support combustion; however, the first consideration must of necessity be its chemical resistance to the corrosive condition.

Thermo-Conductivity

The problem may arise of protecting a piece of equipment which has some form of heat exchange in conjunction with its function. For this reason it may be necessary to choose a protective material which has little or no effect on the heat transfer coefficient of metal surfaces. Conversely, it may be desired to insulate as well as protect. The requirements and the characteristics of the protective materials will dictate the choice. Thermo-conductivity is another factor in the selection of a protective material.

Water Absorption

Water absorption and moisture vapor transmission are two separate things; but for purposes of this discussion are considered together. The reason for this decision is the net end result when moisture passes through the film or is absorbed by the film. In both cases there eventually is a loss in adhesion of the protective material and, consequently, a loss in protection. It must be remembered that water carries dissolved solids, liquid solutions, and gases with it as it is absorbed or as it passes through the coating or covering material. Water and oxygen cause steel to rust and in so doing increase in volume and push the protective material from the surface it is trying to protect. Acidic water attacks both masonry and concrete, again destroying the adhesion of the protective material. Water alone causes wood to swell and separate from its protective cover. As water is generally part of the waste being treated, it becomes mandatory to select a protective material that has excellent water resistance.

Film Thickness

Film thickness varies from manufacturer to manufacturer and from material to material. However, it does not necessarily follow that the thicker the film the better the protective material. Personal experience will verify that a light wool blanket provides better heat insulation than a much heavier cotton one. In similar manner, thin films of some materials give better corrosion protection than much thicker films of other materials. The important thing here is to choose a material which is inert to attack by the wastes or the treating chemicals.

Weather Resistance

Weather resistance implies that part or all of the treating plant is subject to weathering. Outdoors are found sunlight and actinic rays, which tend to

affect adversely some types of coating materials. Variations in the ambient temperature, humidity, and atmospheric contaminants all have an effect on the coating material. Weathering can be, under some conditions, as rough on a protective material as constant immersion in a chemically reactive solution. Change in temperature alone can be responsible for separation of the coating material from the base material of construction. Atmospheric corrosion should not be lightly dismissed, as it can present some well nigh insuperable problems.

Acid Resistance

A large proportion of waste treatment plants are involved in the treatment of acid wastes. For this reason, specific knowledge is required as to the acid resistance of the selected coating material. All protective coatings have some resistance to acid solutions, but the degree of resistance varies. Most coating materials resist weak solutions; a measure of safety may be gained by diluting the wastes with water to such a point that the acid does not attack the coating material. It also helps if the temperature of the waste is kept as low as possible while the treating is going on. However, it does not pay to take chances when treating an acid waste. Here it is mandatory to apply a skin that has maximum resistance to the acid, the water, abrasion, temperature, any solvents that may be present, oils, etc. This requires a sheet material of extreme resistance.

Alkali Resistance

Alkalis have some adverse effects upon materials of construction, but in general take a long time to do their damage, which consists mostly of embrittlement of steel, and digestion of wood. For the most part, then, what is needed is a protective coating material to act as a contamination-proofing material rather than as a protective one. Alkalis, however, have the characteristic of reacting with oils to saponify them and form a soap. Selection of a protective coating material formulated with a saponifiable material is sure to invite trouble if the treatment involves an alkaline waste, or addition of alkalis to neutralize acidic wastes. This generally rules out those coatings based on oils or greases.

Solvent Resistance

Some types of industrial wastes contain varying amounts of solvent materials. These solvents may be of any kind and may dissolve the selected coating material, thus baring the equipment to the action of the wastes. In all cases there should be a check on the possibility of a solvent material being included in the waste to be treated. A coating material which may be dissolved or softened by wastes which include a solvent should never be selected.

Salt Resistance

Corrosiveness of salts on steel and other metals as a result of salt spray tests is probably universally known. Usually these tests are conducted to determine the effectiveness of a coating material in curbing or stopping corrosion. No trouble should be encountered with this factor, as there are protective materials which are unaffected by all concentrations of salts at normal operating temperatures. However, the limitations of the selected coating material should be known, as salts are likely to be developed in or added to the wastes during treatment of them.

Oil Resistance

Some forms of industrial wastes carry oils, which are to be salvaged and returned to use in the industry. These oils oftimes have a detrimental effect upon the protective coating material. They tend to act as a solvent and if they do not dissolve the material, they may soften it to a point which is dangerous. Fortunately, there are coating materials which are immune to the ac-

tion of all oils, whether they be animal, vegetable, or mineral.

Toxicity

The toxicity of any material which may be selected is of passing interest only. This point should be taken into consideration, however, when making the selection, as it may require protection of the application crew and, possibly, clearing the area of other personnel during application.

Special Conditions

There are certain conditions of application which must be taken into consideration when selecting a protective material. For the most part, it would be acceptable to take any one that is satisfactory as to chemical resistance to the waste being processed. However, it becomes mandatory to think in terms of the applied material. At times, it is almost impossible to do a good job of application with a certain type of coating material. Therefore, it becomes necessary to make a second choice. It may be found more effective to redesign the equipment before making a coating selection or to redesign it to accommodate application of the selected material. This point is mentioned to show that there may be some special condition which should be taken into consideration when making a selection.

Application of Coating

As regards application of the protective material, there is just one point to make: be sure that the application is done by the best qualified crew it is possible to obtain. The protective material is only a solution or a sheet until it has been applied to the plant or equipment. How well the surfaces are prepared and how meticulously the application is carried out determine the effectiveness of the finished job. Application is just one of the links in the chain of events—it should not be the weak one.

Economics of the Project

There are still other factors which must be considered before the selection is made, all of which bear upon the economical aspects of the job. The objective should be the selection of a protective material that will give the lowest cost per unit of area per unit of time. To do this, it is necessary to know how long the operation is to run; whether there will be a change in the type of waste being treated that would call for a dual-purpose coating; the possible trouble-free life expectancy of the selected protective material; the relative cost of repairing the selected coating; the cost of repair, with reference to the plant and/or equipment should the protective material fail; whether a protective material is needed on all of the plant and equipment; and the effect on production should the coating material fail. All of these things have a bearing on the quality of the coating material to be selected for protecting the equipment and plant used in the treatment of wastes.

SEWER MAINTENANCE AT MARSHALL, MICH.*

By Victor D. Camp

City Engineer, Marshall, Mich.

The time-worn adage that "an ounce of prevention is worth more than a pound of cure," although quite trite, certainly can be applied to the maintenance necessary for proper functioning of municipal sewer systems. The ounce of prevention is the proper con-

* Presented at 1951 Annual Meeting, Michigan Sewage and Industrial Wastes Assn.; Grand Rapids, Mich.; June 4–6, 1951.

struction of sewer systems in order that the sewers will function properly and require a minimum of maintenance as the years roll by.

Many engineers are too economy minded in the construction of important public works. Although it is most important that construction costs be kept as low as possible, the quality of workmanship and materials incorporated in public works should be of the best, for over-economy simply means that any consequent savings in construction costs will be nullified by the increased maintenance cost of the improvement. This is particularly true with the construction of sewer systems.

The proper design and field engineering of sewers is also of the utmost importance. Specifically, there comes to mind a costly repair job on a section of 8-in. sanitary sewer where about 30 ft. of standard vitrified pipe was found in a collapsed condition. This section of sewer was located in a deep cut and had caused no end of trouble from sewer stoppages. It finally became necessary to excavate down to the pipe to remedy the trouble and, as suspected, the pipe had collapsed under the weight of the backfill. The sewer was more than 20 ft. deep at this location and the original sewer builders neglected to use extra-strength pipe to compensate for the extreme weight of the backfill in the deep trench.

Engineers now have much better products and materials to incorporate in sewer construction than were available 20 or more years ago, thanks to progressive material manufacturers and the demands of the engineering profession. If the materials are properly incorporated in the sewer construction and if the sewer is properly designed, there need be but little concern over future maintenance of the system.

Anyone who is responsible for the maintenance of sewers will agree that tree roots are by far the greatest source of trouble. Proper construction practices using modern jointing materials should eliminate almost all trouble from this source. Here again, in spite of the availability of superior products, sometimes inferior joint construction results because of poor inspection and questionable construction methods. There is no excuse for any sewer constructed under ordinary conditions to be susceptible to root stoppages at a later date.

Ideal Not Always Attainable

In the matter of design, there is an ideal to be striven for. Unfortunately, however, circumstances beyond control, such as adverse topography, location, economic restrictions, and other restricting factors, limit the design to less than the ideal for the proper functioning of the system. With future maintenance in mind, it should always be the designer's goal to provide sewer gradients that will provide enough velocity to the sewage to prevent settling of solids in the sewer. The design of surface sewer accessories, such as curb inlets and catch basins, should provide adequate settling space for the collection of silt, sand, and gravel. The entrance of this heavy material into the sewer pipe will create a costly maintenance job of removal. The removal of this foreign material from the drainage structures is an item that should not be neglected or the sewer pipe will be plugged by the material overflowing the basins.

House Sewer Connection a Weak Point

Another phase of municipal control that is very important in reducing sewer maintenance is the proper inspection and control of lateral connections to the sewer proper. Plumber's connections to the sewer pipe wyes and tees cause much trouble between the city officials and the property owners. On numerous occasions a "post mortem" on a house connection has shown root growths entering the system at the

point of connection of the house lateral to the public sewer. There is little wonder that such is the case after watching some plumbers make these connections. Their attitude seems to be: "If I can make this tap without getting buried in the trench, I will be all set." Making the tap to the sewer is the most dangerous and disagreeable part of the plumber's job of running a house connection in from the street. Many plumbers are poorly equipped for this work if done under adverse conditions. A tough plumbing inspector can do a great service by keeping a strict watch on all sewer connections to see that they are properly made, properly supported, and properly backfilled.

The matter of house connections brings up an interesting interpretation of responsibilities. For instance, the Grand Rapids sewer ordinance specifically states that the lateral from the building to the city sewer is the responsibility of the property owner. And the ordinance states also that the plumber's connection shall be inspected by a city agent to see that it is properly constructed. The "$64 question" has been put at numerous times by irate property owners when they have to dig up or re-lay private sewers. They often find that their stoppage was caused by roots entering at the plumber's connection to the main sewer. The question is, of course, "Why shouldn't the city pay for this repair job?" To date, the city has not paid any of these so-called claims. However, the author knows of one citizen who says he will sue the city the next time his sewer is plugged by this kind of a stoppage.

So much for the "ounce of prevention" previously alluded to. Inasmuch as many mistakes of the past as well as the present are inherited, the matter of maintenance is always present.

Maintenance Program

An alert and efficient maintenance crew is the best remedy for the headaches occasioned by wrathful citizens with flooded basements. By alertness is meant having men ready at all times of the day or night to handle the wet and filthy equipment necessary to unplug choked up sewers. To be efficient, the proper equipment and personnel must be provided to cope with the situation.

Two trends of practice seem to prevail in the application of maintenance methods to sewer systems. A large percentage of municipalities schedule a regular program for the inspection and cleaning of sewers. Others, particularly the smaller cities and villages with restricted budgets and a small public works department, seem to more or less control the situation by waiting for sewer trouble to come rather than going out looking for it.

Many years ago, when the author first went to work for the city of Marshall, a three-man crew was sent out in the springtime each year to inspect and flush the entire system, with emphasis on the dead-end sewer terminals. By this flushing process, large root growths were detected by the backing up of the sewage. The roots were removed as completely as possible with the tools available. In spite of this yearly inspection, there were many unexpected sewer stoppages. Calls were received in the middle of the night and on Sundays and holidays, as well as during the working day, to assist someone distressed by a flooded basement. As time went on, a definite pattern of inspection became apparent as it was found that certain locations in the system gave considerable trouble and the rest of the system gave little or no trouble. As a result, the city-wide system of inspection and flushing was discontinued and the crew's efforts were concentrated on those sections of sewer causing the most trouble. This practice has now been discontinued, due to lack of help and maintenance funds, but quick emergency service is still provided at all times by experienced men equipped with modern maintenance equipment.

For sewer cleaning, a flat-bed truck, carrying a complete line of Flexible steel rods and accessory tools actuated by a power unit, is provided. This equipment has proven very satisfactory, as it is no longer necessary for the men to crawl down into unsafe and messy manholes to manipulate sewer rods manually. Also, the present equipment is much faster and more efficient in removing root obstructions.

After taking care of an emergency, it is established policy to thoroughly inspect and clean that section of the sewer which has caused the trouble. This inspection is repeated at various intervals to be sure that the section of sewer is functioning properly.

Root Reduction Experiments

It may be of interest to know that flake calcium chloride and rock salt are being used experimentally in sewers that have a bad root history. This is applied in similar manner to applications of copper sulfate, except in larger doses. From one-half bag to a bag of chloride is dumped in a manhole, followed by a 50-gal. drum of water dumped from a truck. The resulting brine solution, contacting the fine fibrous live roots, does the trick. Conclusive results of the experiments are not available and this may be common practice elsewhere. However, evidence indicates that it helps kill the roots. An 8-in. lateral sewer on Monroe St. gave continual root trouble until a large pickle company moved to town. As soon as they started dumping the contents from their salting vats into this sewer, root trouble disappeared. On one occasion inspection of a house lateral that was dug up showed the roots to be dead for at least 6-in. outside the pipe. The owner had been using salt in his sewer at the author's suggestion.

All who are in any way responsible for the efficient operation of sewer systems know that the maintenance of the sewers is a very undesirable and disagreeable job. But it is a job that is imperative to the health and welfare of the community's citizens and sewer departments must give their very best attention to this most important municipal function.

EFFECTS OF PENICILLIN WASTES IN LEY CREEK SEWAGE TREATMENT PLANT, SYRACUSE, N. Y.*

By Uhl T. Mann

Superintendent, Ley Creek Sewage Treatment Plant, Syracuse, N. Y.

Sewage works literature contains few data on the treatment of penicillin wastes. This is probably due to the fact that the large-scale production of this product is a relatively new industry.

The deep-vat method, which brought about large-scale production and the resultant large quantities of wastes, was first used in about 1943. Since 1943, the size of the deep vats has increased from about 1,200 gal. to the present size of 30,000 gal. Each step has increased the quantity of waste to be handled by treatment processes.

Several unpublished articles had pointed out that penicillin wastes had been treated by biological processes. In several instances, however the information was contradictory. In one case, aerobic stabilization was successful, followed by unsuccessful attempts with anaerobic digestion. In other localities digestion was successful, whereas wastes fouled various types of filters and the activated sludge process.

* Presented at 1951 Joint Meeting, New York Sewage and Industrial Wastes Assn. and New England Sewage Works Assn.; Springfield, Mass.; June 7–8, 1951.

Hilgart (1) reported successful treatment of liquid wastes on trickling filters and digestion of solids. It is well to point out that the same article reports difficulty in establishing normal digestion. Muss (2) reported successful treatment of penicillin wastes on several combinations of units. The same study pointed out that wastes from penicillin readily respond to almost all methods of treatment when highly specialized operating procedures are employed. The addition of sanitary sewage to the industrial waste is necessary to provide the proper microorganisms for biological treatment. Rudolfs (3) has reported successful treatment both with and without addition of sanitary sewage. Brown (4) also reported successful treatment of penicillin wastes in conjunction with other pharmaceutical wastes.

Nature of Wastes

Penicillin is produced by the deep-vat method. The culture medium for growth of the mold is corn steep liquor, to which lactose and certain mineral salts are added. The medium is inoculated with a culture of the mold. The fermenters (Figure 1) are continuously agitated by compressed air and are kept at a warm temperature to promote the growth of the mold. At the time of harvesting the ferment liquor is removed from the tank, the growth inhibited with formaldehyde, and the mold is removed by vacuum filtration and discarded. Next, the broth is acidified and extracted with amyl acetate. The amyl acetate extract is separated from the medium by centrifuge and in turn is extracted with a buffered salt solution. Purification of the penicillin is accomplished by further treatment with solvents. The solvents are removed by distillation for re-use.

The wastes are made up of the solid penicillin growth, which is highly nitrogenous and has a high B.O.D. The spent broth is high in B.O.D. and suspended solids and contains proteins, sugars, and organic acids. The waste process water contains waste acid and caustic materials, as well as the unrecovered solvents.

In addition to the manufacturing wastes these plants have the problem of disposing of contaminated batches, which are high in both suspended solids and B.O.D. For the present purposes it will suffice to refer to the articles by Hilgart (1) and Muss (2), which mention B.O.D. running as high as 30,000 p.p.m. with suspended solids reaching 20,000 p.p.m. In the early experiences at the Ley Creek plant B.O.D. and suspended solids values as high as 35,000 p.p.m. have been recorded on samples taken at the penicillin plant sewer. The latter value represented waste before the mat was removed and before any stripping operation. Under present operations B.O.D.'s and suspended solids concentrations have been reduced to 5,000 and 3,000 p.p.m., respectively. Apparently, the strengths of penicillin wastes vary with different manufacturers. To a large extent the B.O.D. load will be directly related to the success of the stripping operation.

In general, penicillin wastes are strong and there is considerable variation in strength between batches and even between days. In plants using the solvent amyl acetate there is always the possibility of an interruption in the stripping operation to remove the solvent. Amyl acetate, which has a B.O.D. equal to its own weight, can impose a staggering load on any sewage treatment plant if received in appreciable amounts.

Ley Creek Experience

The Ley Creek plant in Syracuse, which is a conventional activated sludge plant, has been treating penicillin waste mixed with the normal sewage flow since May 1945. Experience at the Ley Creek plant showed that it was not the nature of the wastes

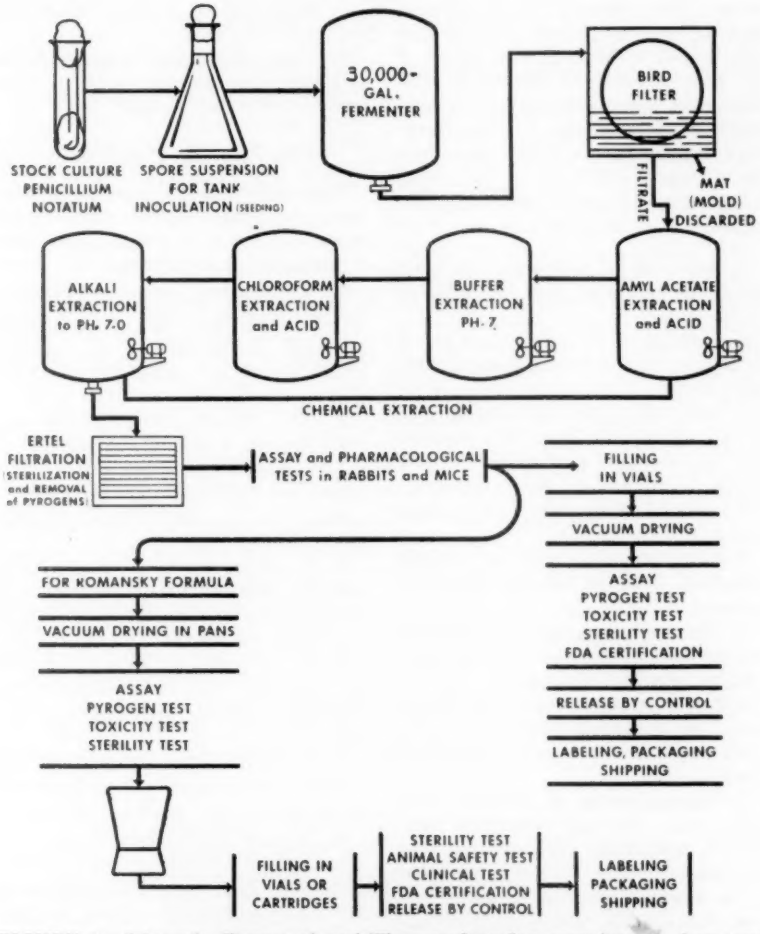

FIGURE 1.—Schematic diagram of penicillin manufacturing operations to show types and sources of wastes produced.

which interferred with operation, but the quantities. If the quantity of penicillin waste added to the sewage did not increase the plant loadings beyond design basis, results were satisfactory. In fact, aeration tank loadings reached as high as 125 lb. of B.O.D. per 1,000 cu. ft. of tank capacity (two to three times normal loadings) without knocking out the activated sludge process.

By nature, these wastes are high in organic material that does not respond readily to primary settling and is passed on to aeration treatment. A like amount of solids after activated sludge treatment occupies many times the volume that it would have as primary sludge. This imposes a volume load on digesters, even though loadings on the basis of volatile solids may be normal.

There were early troubles at the Ley Creek plant with the new waste ma-

terial. This, however, is no different than any new operation. The troubles were solved by mutual cooperation of the New York State Health Department, the manufacturers, the manufacturer's consultants, and the Onondaga Public Works Commission and its advisors.

Corrective measures taken by the industry were as follows:

1. Removal of formaldehyde from wastes to eliminate future inhibiting of digestion.
2. Installation of a stripping tower to remove the solvents, particularly amyl acetate.
3. Removal of mat waste from flow to the sewer. At first this material was hauled away as a slurry in tank trucks. Later the mat waste was dried and disposed of by dumping on farm land.
4. Elimination of sources of possible leakage of material used in manufacturing.
5. Thorough program of sampling and testing for solvents in flow leaving the manufacturing plant.
6. Careful programing of the dumping of contaminated tanks to prevent shock load at the sewage treatment plant.
7. Neutralization of sulfuric acid waste with calcium hydroxide sufficient to protect the sewer and prevent interference with biological processes at the sewage treatment plant.
8. Installation (at the manufacturer's expense) of a sludge lagoon to supplement the drying bed capacity.

The foregoing remedial measures were taken as the needs developed. At the same time the Ley Creek plant used its facilities to handle a plant overload in the best possible manner. Expedients used in attempts to improve operation were as follows:

1. Higher rates of recirculation of activated sludge.
2. Chlorination of return sludge.
3. Recirculation of final effluent to reduce shock loads.
4. Cross pumping of aeration tanks to provide the aerators with the best possible activated sludge.

Summary and Conclusions

The experience of operation with penicillin wastes mixed with sewage at the Ley Creek treatment plant may be summed up as follows:

1. Penicillin waste is amenable to aerobic and anaerobic stabilization, provided no substances are present which poison or retard biological activities.
2. The manufacturer should use every known pretreatment method to reduce organic load going to the sewer. The quantity of the waste, and not its nature, will seriously affect the sewage treatment plant.
3. Aeration treatment of penicillin wastes produces larger volumes of sludge than are normally produced with domestic sewage.
4. The presence of solvents imposes an additional load on aeration units, with the resulting additional cost for air.
5. The manufacturer should provide some means to smooth out and prevent shock loads from reaching the sewer.
6. It is deemed advisable for the manufacturer to inform the proper authorities with regard to changes in the manufacturing operation, so that careful consideration can be given to the resulting effect on the sewage plant.

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TIPS AND QUIPS

Wine 'Em and Dine 'Em

If your plant is plagued with rats, as so many are, don't discourage them openly. Instead, be the gracious host and provide them with food and drink —served from a special "cafeteria" of their own. Of course, the food supplied will contain warfarin, the potent raticide chemical discovered at the University of Wisconsin, and the "wine" will be that priceless beverage known as "Poor Man's Champagne" (water), which is so important in rodent poisoning operations.

To make the chores of such hospitality easier for the host, the Solvit Chemical Company, Madison, Wis., has introduced a large-size, portable, weatherproof steel bait container designed especially for use with warfarin rat baits. The manufacturer claims that highly effective control of rat infestations, even in open public dumps, is made possible with the Rat Cafeteria (Figure 1), which holds enough bait for 60 to 90 days without replenishing. Integral provision of readily available water is said to make the cafeteria more attractive to its potential patrons.

Flies Becoming Immune to DDT

Domestic flies all over the world are becoming immune to DDT, according to an article in a recent issue of the *South African Standards Bulletin*. It is suggested, therefore, that the best way to get even with them is to use different insecticides in rotation so as to minimize the flies' resistance to DDT.

A DDT-resistant strain of house flies has been developed, by selective breeding from survivors of successive generations subjected to DDT spraying, by two British investigators. Also, prolonged routine tests carried out in the entomological laboratories of the South African Bureau of Standards indicated that DDT-resistant flies have likewise been found in that country.

Costs Reduced by Chrome Plating

Practical application of the extreme hardness of electrically deposited chromium has been made by W. W. Mathews, superintendent of the Gary (Ind.) Sanitary District. Reporting on the matter in his 1950 annual report, Mr. Mathews says:

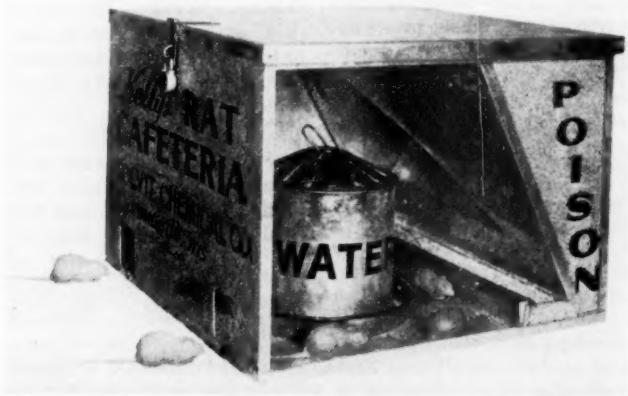

FIGURE 1.—Rat "cafeteria" for use with warfarin rat baits needs attention at infrequent intervals and puts bait and water within easy reach for effective rat control.

"There are five gas engines in the Gary plant. The piston pins on these engines wear elliptically and have to be replaced. Instead of replacement, the old pins are machined round and then chrome plated to their original diameter. The saving is just over $30 per pin.

"Raw sludge is pumped to the digesters with direct-acting simplex pumps. Wear on the plungers of these pumps is excessive because of the fine grit carried over in the raw sludge. The plungers are 9 in. by 12½ in. and made of cast iron. Replacements were required at short intervals. Much of this replacement requirement was eliminated, however, by chrome plating new plungers and machining and chrome plating the old ones. Savings by chrome plating amount to $35 per plunger."

Bucking the Bureaucracy

Stream pollution authorities, especially in Maine, have found themselves forced to take more stringent enforcement steps in a direction generally unheard of until federal government agencies began outdoing themselves in seeing which could spend the most money to undo the other's work.

The medium in the Maine instance is the lowly potato. To hold prices up, one federal agency buys and dumps surplus potatoes, despite the attempts of another federal agency to hold prices down and prevent inflation. This in itself wouldn't bother the stream pollution people—except, perhaps, in the region of the pocketbook. But the dumping of surplus potatoes on the banks of and into inland streams in Aroostook County during years of excessive potato production resulted in such a stream pollution problem, due to their decomposition, that legislation was enacted in 1947 prohibiting such practice. In that year, according to an item in the August, 1951, *News Letter* of the New England Interstate Water Pollution Control Commission, some 10,000,000 bushels were dumped, and 73 dumps were found to be located on the banks of rivers and streams. A surplus potato crop in 1949 resulted in some illegal dumping. To strengthen the law by providing easier enforcement, the 1951 Legislature passed amending legislation.

Now a new problem in connection with waste potato dumps has arisen, in that the dumps are found to be prolific breeding places for the *Drosophila* fly (commonly called "fruit flies"). However, that's another agency's problem and the merry-go-round continues!

For That Icy Spot

An improved type of Melt, ice and snow-melting chemical pellets reputed to have 30 times the thawing capacity of salt, has been announced by The Chem Industrial Co., 1114 Hippodrome Bldg., Cleveland 14, Ohio. The new product, 99 per cent active chemically, is claimed to produce greater exothermic action than previously offered material. The manufacturer advises that Melt's ice and snow-melting ability is approximately 10 times that of flake calcium chloride. The material is applied by sprinkling sparingly over ice or snow surfaces. It has great covering capacity and, if used at the start of a snow or sleet storm, will melt subsequent snow falls. The manufacturer recommends it for use on streets, driveways, sidewalks, parking lots, loading platforms, steps, etc., and for thawing frozen gutters, pipes, sewers, and railway switches. A handful under rear car wheels will provide traction and stop wheel spinning. There is no messy residue.

Melt is available in 25-lb. metal pails, 100-lb. fiber drums, and ton lots packed in 100-lb. moisture-proof paper bags.

Reviews and Abstracts*

National Committee on Radiation Protection. By J. H. Jenson. *Ind. Eng. Chem.*, **43**, 1500 (1951).

The subcommittee on waste disposal and decontamination is one of eight working subcommittees serving under the National Committee on Radiation Protection. This subcommittee strives to serve as a clearing house for problems and recommendations dealing with the disposal of radioactive wastes and the decontamination of laboratory surfaces and equipment.

The subcommittee has circulars in preparation and is actively working to obtain data for other circulars which should be of help in laboratories. The subcommittee will welcome suggestions on waste disposal. It does not want to recommend procedures which will work undue hardships, but the recommendations must assure safe, sane handling of radioactive materials.

R. S. Ingols

Liquid Waste Disposal at Oak Ridge National Laboratory. By F. N. Browder. *Ind. Eng. Chem.*, **43**, 1502 (1951).

The Oak Ridge National Laboratories have a wide variety of radioactive wastes in large volumes of water. The liquid must be discharged into a small stream wholly within the guarded area at Oak Ridge. This stream can be used for study and close control. The small stream enters into the Clinch River at the edge of the guarded area. The stream is dammed in several places to increase the storage time.

The radioactive wastes are gathered into a surge tank and then admitted to an evaporator. The condensed vapor is placed in a settling basin and then goes to the river after monitoring. The concentrated residue is placed in storage until it has "cooled off" enough to be discharged safely or permanently buried. This removes most of the radiation from the natural streams, but obviously very large volumes of water thought to be free of contamination go directly to the stream. A large amount of activity reaches the stream in this manner. Its source and correction must be accomplished.

Progress is being made toward solving the present difficulties of the Oak Ridge National Laboratories to minimize the discharge of radioactivity to the natural drainage system.

R. S. Ingols

Concentration of Radioactive Liquid Waste by Evaporation. By G. E. McCullough. *Ind. Eng. Chem.*, **43**, 1505 (1951).

The radioactive contaminated liquid from the Knolls Atomic Power Laboratory is disposed of in a separate building in the area. The liquid is treated with caustic and then introduced into a flash evaporator under vacuum. The vapor passes through a separator column and is then condensed and placed in a temporary storage tank. The liquor is monitored and pumped to the sewer unless some carry-over is detected. The concentrated radioactivity is transferred to vacuum tray driers and then into large steel drums. These are stored until "cold" or finally disposed of.

R. S. Ingols

Removal of Plutonium from Laboratory Wastes. By C. W. Christenson, M. B. Ettinger, G. C. Robeck, E. R. Hermann, K. C. Kohr, and J. F. Newell. *Ind. Eng. Chem.*, **43**, 1509 (1951).

A study of the removal of plutonium from laboratory wastes was undertaken in

* Please send to Federation headquarters all periodicals, bulletins, special reports, etc., which might be suitable for abstracting in This Journal. Publications of public health departments, stream pollution control agencies, research organizations, and education institutions are particularly desired. Address such material: Federation of Sewage and Industrial Wastes Assns., 325 Illinois Bldg., Champaign, Ill.

order to secure information upon which to base pilot plant design. Results are presented from investigations which included: coprecipitation with iron, aluminum, and other metal ions; adsorption by various agents such as activated carbon, celite, kaolin, etc.; and removal by activated sludge.

The activated sludge process required a three-stage counter-current plant with the addition of organic food sources ahead of each stage in order to reduce the plutonium content of the effluent to the desired level. Activated carbon was the best of the adsorption agents studied, but would require the disposal of large amounts of radioactive sludge unless split treatment with long periods of mechanical agitation were used.

In the absence of substantial amounts of complexing agents, the coprecipitation with an iron floc appeared to be the simplest, cheapest, and most efficient method. Aluminum could be used, but was not as satisfactory. Only 10 p.p.m. ferric chloride with sufficient lime to keep the pH above 7.0 was adequate to produce the necessary plutonium removal.

A treatment plant was constructed to provide prolonged flocculation and sedimentation or serial flocculation and sedimentation and low filtration rates. Results of two months' operation of the pilot plant confirm laboratory findings. Sludge recirculation appears to give some improvement.

Because of the high fluoride content of the wastes, fluoride removal is required to protect ground waters. Fluorex filters were found to be successful in fluoride removal.

R. S. INGOLS

Laboratory Studies on Removal of Plutonium from Laundry Wastes. By J. F. NEWELL, C. W. CHRISTENSON, E. R. MATHEWS, C. C. RUCHHOFT, H. L. KRIEGER, AND D. W. MOELLER. *Ind. Eng. Chem.*, **43**, 1516 (1951).

Because of the health hazards of handling radioactive material, all laundry from an area where it is known that the soiled clothing is or may be contaminated with radioactive material must be handled by personnel trained for the work. Thus, each major A.E.C. project has its own laundry where gowns and slippers of all exposed personnel may be processed. Since a gown must be monitored after every use of radioactive material and "hot" ones discarded, several gowns may be used in one day by one person. During the war, security reasons necessitated the laundering of the equipment within the classified area.

Because of the use of detergents in the laundry, studies were made on the treatment of the wastes from the laundry at Los Alamos which were separate from those of the laboratory and reported by Christenson (see above). The wastes contained soap, detergents, and citric acid, as well as lint. The 5-day B.O.D. values of the waste varied from about 200 to 600 p.p.m. The alpha radiation activity from plutonium was relatively low (in comparison to the normal plant activity or that from the laboratory waste itself), but averaged about 1,500 counts per minute per liter, although it rose to maxima of 20,000 counts per minute per liter. It was necessary to reduce the plutonium activity in the effluent to 70 counts per minute per liter or less. A reduction in the B.O.D. was desirable, but of secondary importance.

Experiments indicated that biological treatment was superior to chemical treatment because carrier precipitation encountered difficulties from the effect of the complexing agents needed to remove the active plutonium. Activated sludge treatment was not feasible on detergent wastes because of excessive foaming. A trickling filter requires a very high ratio of recirculation (15 to 1 or better) with almost complete nitrification to obtain satisfactory removal of the plutonium. Even then, ammonia and phosphate may have to be supplied to the waste before treatment in order to get the desired growth. The volume of sludge from the trickling filter is adequate for disposing of the radioactive material, but is only 1/25 the volume of sludge produced by carrier (chemical) precipitation.

The pilot plant was to be designed to have two-stage filters with capacities for varying but high recirculation ratios and low loading of the primary filter on a B.O.D. basis. It may also be necessary to have a sand filter after the second stage to prevent any sludge from going into the final effluent.

R. S. INGOLS

Activated Sludge from Foods for Treatment of Radioactive Waste. By C. C. Ruchhoft, F. I. Norris, and L. R. Setter. *Ind. Eng. Chem.*, **43**, 1520 (1951).

In the treatment of an industrial waste containing radioactive material by the biological method of activated sludge, it is essential that the activated sludge be produced in sufficient quantity to provide an excess for the daily wasting of the sludge. This is necessary, in order to keep the concentration of radioactivity at a reasonable level.

Experiments were conducted to evaluate the possibility of using common foods plus mineral supplements for the production of activated sludge where the nontoxic, inorganic industrial waste was to be treated. It was concluded that common foods and essential minerals can maintain good control of the activated sludge process.

R. S. Ingols

Hoffman-La Roche Exhibits Waste Disposal Facilities. Anon. *Chem. Eng. News*, **29**, 2468 (1951).

A solution to the problem of waste disposal of a producer of complex organic chemicals was shown to a group of chemical engineers at an open house. Waste disposal is a problem because of the substantial amount of acids, alkalies, and solvents used by the plant. Wastes are first taken to a storm sewer and then to a settling basin. Provision is made for automatic pH control and for diversion of the treated storm sewage to the Passaic Valley (N. J.) sewer system.

R. S. Ingols

Flow Colorimeter Adapted to Chlorine Analysis. By R. C. Hawes, R. R. Davis, H. H. Cary, and A. O. Beckman. *Anal. Chem.* **23**, 503 (1951).

An instrument is described which is capable of recording the quantity of chlorine in a flowing air-gas mixture. The special precautions for handling the corrosive factors of the chlorine are discussed. The method for maintaining the instrument in proper calibration in spite of power line voltage fluctuations is given. Ultraviolet light is used for measuring the chlorine.

R. S. Ingols

Determination of Fluorine in Monofluorinated Organic Compounds in Air and Water. By J. M. Salisbury, J. W. Cole, Jr., L. G. Overholser, A. R. Armstrong, and J. H. Yoe. *Anal. Chem.*, **23**, 604 (1951).

Widespread use of rodent poisons containing sodium monofluoro acetate or similar compounds presents the problem of identifying the organic compound in dilute water solution. In addition, widespread manufacture of these compounds presents the possibility of their being discharged in an industrial waste. According to this article, the analysis is carried out only for the quantity of the organic fluoride in the sample; no identification of the organic fraction of the various compounds is indicated.

The authors recommend the conversion of the organic fluoride compound to the free fluoride ion by refluxing the solution with a mixture of potassium metaperiodate, silver perchlorate, perchloric acid, and glass wool. The silicon tetrafluoride was broken down and the fluoride removed from the complex mixture by distillation according to standard procedures. The fluoride ion in the distillate was determined colorimetrically.

R. S. Ingols

Titration of Dissolved Oxygen Using Acid-Chromous Reagent. By H. W. Stone and R. L. Eichelberger. *Anal. Chem.*, **23**, 868 (1951).

The authors have adapted the chemicals used in the technique of gas analysis to the problem of determining dissolved oxygen in very small samples of water. The chromous ion in the presence of hydrogen ions will react quantitatively with molecular oxygen to form chromic ion and water. By adding a sample of water to the acid chromous reagent under a blanket of inert gas, the dissolved oxygen is allowed to react. The excess chromous ion is then determined by adding a standard solution of potassium iodate and back titrating the excess iodate in the presence of added potassium iodide and starch with more of the original acid-chromous ion reagent.

Only 5 ml. of sample are needed, although sample size is apparently not critical. The reagents must be very carefully prepared and stored in special

burettes to be free of oxygen. A magnetic stirrer is used for mixing the sample with the reagents and 400 ml. of carbon dioxide gas is used per minute to keep the reaction vessel free from gaseous oxygen.

The method gives good reproducability and accuracy in the hands of the authors, who recommend it for problems involving water supplies and waste waters. They indicate, however, that nitrites do interfere somewhat in the determination. They do not show the effects of any interfering organic substances, such as tannins, for its application to industrial wastes.

R. S. INGOLS

Colorimetric Estimation of Various Metal Derivatives of Sodium Diethyldithiocarbamate. By R. J. LACOSTA, M. H. EARING, AND S. E. WIBERLEY. *Anal. Chem.*, **23**, 871 (1951).

The organic reagent, sodium diethyldithiocarbamate, has been widely used for the colorimetric determination of copper. However, 1,000 p.p.m. of bismuth, cobalt, chromium, iron, nickel, and uranium, as well as copper, can be determined colorimetrically using this reagent and subsequent extraction into a chloroform solution. The extraction of the colors is quantitative over a wide range of pH. All of the elements listed will interfere in the determination of copper unless removed before developing the color. The various elements give somewhat different maxima of light adsorption so that readings at each maximum for each of any two elements known to be present should permit the determination of the amount of each present. A control of pH during development of the color will aid in separating copper from uranium and chromium.

R. S. INGOLS

Microdetermination of Arsenic and its Application to Biological Material. By G. R. KINGSLEY AND R. R. SHAFFERT. *Anal. Chem.*, **23**, 914 (1951).

A special all-glass apparatus has been developed to handle an accurate and sensitive method for the determination of less than 0.1 microgram of arsenic in biological material. Samples up to 25 ml. in volume are digested with hydrochloric acid for several minutes. Iodide and stannous chloride are added to reduce the arsenic. Using the special apparatus, zinc is added so that arsine is generated and distilled into a dilute iodine solution. Color reagents of ammonium molybdate and hydrozine are added and the color intensity determined by visual or photocolorimetric equipment. Hydrogen sulfide must be removed from the gas stream during distillation.

The test has been designed for biological material, but should work equally well with pertinent industrial wastes.

R. S. INGOLS

Ultraviolet Spectrophotometric Determination of Vanadium. By G. TELEP AND D. F. BOLTZ. *Anal. Chem.*, **23**, 901 (1951).

A method is described of determining vanadium in solution by an oxidation with hydrogen peroxide. The compound, of reddish-brown color, also adsorbs strongly at 290 millimicrons with less interference from other metals. The test is simple and rapid.

R. S. INGOLS

Small Unit Converts Sewage to Pure Water. ANON. *Chem. Eng. News*, **29**, 3016 (1951).

It has been announced by the California Institute of Technology that a filter can convert sewage into potable water at the rate of 2 qt. per min. for 30 to 40 min. There is a mechanical strainer on the intake and a chemical filter to remove the fine suspended matter and kill the bacteria

R. S. INGOLS

Third Annual Review of Analytical Chemistry. Reprint from *Anal. Chem.*, **23**, pp. 1–101 and 212–257 (1951).

This review includes 18 articles on the techniques of analysis in the January issue and 8 articles on the various materials analyzed in the February issue. In this group S. K. Love of the U. S. Geological Survey has a review of 101 articles on the analysis of water (water, sewage, and industrial waste). The reprint is available from the reprint section of the American Chemical Society at $1.50.

R. S. INGOLS

Melbourne and Metropolitan Board of Works, Report for Year ended June 30, 1950.

This Board is composed of Melbourne and 26 cities, with 4 shires. It was constituted in 1890, and deals with water supply, main and general sewerage, drains, and main drainage works. There has been considerable activity on extension of the water supply, the sewerage, and main drainage works during the past year. An area is eligible for sewerage facilities when its population exceeds four persons per acre.

The sewage, including industrial wastes, from about 95 per cent of the population, is treated at the Metropolitan farm, now 26,056 acres in extent, twenty-four miles from Melbourne. Average flow for the year was 66 m.g.d. (Imp.). The principal source of revenue is from sale of cattle and also from sheep. 4,489 head of cattle were sold in the year. On June 30, 1950, there were 13,125 cattle on the farm. Government regulations were modified to permit sale of cattle for slaughter for human consumption after inspection. 3,391 acres are being prepared for irrigation. 504 men were employed on the farm.

Trade wastes, which amount to 22.5 per cent of the daily dry weather flow of sewage, are subject to a charge, which produced during the first year £131,084 ($293,628).

The total staff includes 4,234 employees.

LANGDON PEARSE

The Agricultural Use of Sewage Sludge and Sludge Composts. Tech. Communication No. 7. Memo. by Agric. Res. Council Conf. on Sewage Sludge and Composts. Ministry of Agriculture and Fisheries, Great Britain. Mimeo pamphlet (6½ × 8 in.), 18 pp. (Oct., 1948).

This memorandum summarizes about eight years of field work and collateral investigations at the University of Reading and Rothamsted. The physical properties and chemical composition of sewage sludges are discussed. Composts of straw and sewage sludge were also considered. Field experiments have been conducted since 1942, at first on potatoes and later on market garden crops, using no organic, sewage sludge only, sludge-straw compost, and farmyard manure, with and without N, P, and K fertilizers. On potatoes, the NPK fertilizers alone have larger yields than sewage sludge or compost alone. With manure and NPK, the highest yields were obtained.

From field work on other crops (such as mangolds, sugar beets, and cabbages), the conclusion was reached that sewage sludge is essentially a nitrogen manure, lacking other properties of merit which manure has.

The conclusions are:

1. Sewage sludge has a moderate manurial value as a source of slowly available nitrogen and phosphate. It provides very little potash. In general the crop-producing power of sludge taken from drying beds is much less than that of an equal weight of farmyard manure. The physical effects of sewage sludge on the soil are less pronounced and of a different kind from those of farmyard manure because sewage sludge lacks the coarse fibrous ingredients derived from straw and other plant residues.

2. Only sludges in a comparatively dry state (less than about 50 per cent moisture) are convenient for transport and spreading. As they must be applied at heavy rates and are sometimes difficult and unpleasant to distribute, their use must depend on economic and local considerations so that no general statement can be made on their agricultural value.

3. Digestion improves the physical condition of sludge and the availability of its nitrogen. These improvements are still more evident when the digested sludge is shed-dried or mechanically dried and pulverized.

4. Certain sludges from sewages of industrial origin contain metallic and other wastes, which may render their phosphate unavailable and may sometimes be harmful to crops in heavy dressing. Such sludges, and also those to which iron salts have been added in the course of treatment at the sewage works, should be tested on a small scale before they are used extensively.

5. Satisfactory rotting of straw takes place in compost heaps prepared with about 1.5 parts of sludge dry matter to 1 part of straw, provided the heap is satisfactorily wetted and aerated. Field trials have shown that such composts are better than sewage sludge alone, because they

supply some potash and have better physical effects on the land. Although composts of sludge and straw have proved somewhat inferior to equal quantities of farmyard manure in field trials, they provide convenient methods of adding bulky organic manures to market-garden soils. Only a limited number of field experiments have been carried out on composts of sewage sludge and town refuse; some of these have given useful results.

6. No instance is known in which the use of sewage sludge as a manure for a crop for human consumption has led to an outbreak of typhoid or dysentery infection, but special precautions should be taken in using sludge for salad and other crops to be eaten raw. Wet sewage sludges should never be used for such crops, and sewage sludges from drying beds only when they can be applied to the land some months before these crops are to be sown.

LANGDON PEARSE

North Shore (Ill.) Sanitary District—Report on a Comprehensive Plan for Sewerage and Sewage Disposal. Greeley and Hansen, 149 pp. mss. (Feb. 2, 1951).

In this report, sewerage and sewage disposal is discussed for a sanitary district in Lake County, Ill., fronting on Lake Michigan for a distance of 23.5 mi. from Cook County to the state line, extending inland from 3 to 4 mi. The estimated population in 1970 is 124,000, with a sewage flow of 20.785 m.g.d., containing 22,258 lb. B.O.D. and 22,417 lb. of suspended solids. If the use of household garbage grinders is extended, the suspended solids may be increased by 5,330 lb. in 1970.

Part of the existing sewage effluent enters Lake Michigan; the balance goes westerly to the North Branch of the Chicago River.

The report concludes that the removal of all sewage effluents from Lake Michigan cannot be justified from economic or engineering considerations.

The report recommends abandonment of certain small existing treatment plants, delivery of small flows to the larger plants, and extension of certain submerged sewer outfalls. During the bathing season, all effluents discharged to Lake Michigan should be chlorinated.

The cost of the recommended project is estimated at $4,077,500, with a total annual cost of $448,055. The objectives of the program are: to protect the public water supplies and bathing beaches; to avoid the creation of a nuisance in residential and recreational areas; to make sewage disposal service available ultimately for all areas within the present boundaries of the District.

The various waterworks on the North Shore are provided with modern water filtration plants.

The District now operates 12 sewage treatment works. The southern part of the District is primarily residential. The northerly portion contains a number of large industrial plants.

LANGDON PEARSE

California Pollution Control Legislation. BY L. W. GRAYSON. *Jour.*, Am. Water Works Assn., **42**, 12, 1133 (1950).

This is a review of the events leading up to passage of the 1949 Water Pollution Act by the state legislature, and the subsequent progress in implimenting it. The State Department of Public Health is confined in its activities to water contamination and its abatement, specifically defined as those impairments of water quality where an actual hazard to public health is created.

Water pollution matters come under a newly created State Water Pollution Control Board and 9 Regional Water Pollution Control Boards, the latter conforming generally to the major water basins of the state.

Additional bills passed in 1949 provide financial assistance to municipalities and districts by loans up to the full cost of sewage projects, if need be. In addition, the California Division of Water Resources is directed to make a continuous study of water quality and possible waste-water reclamation in the state.

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There are two batteries of five digesters with a control building for each group, housing a total of eight P.F.T. sludge gas fired No. 1500 Digester Heaters and Heat Exchangers. These heaters are equipped with complete automatic controls to provide constant digester temperatures and heating of the control buildings. Total heater output capacity is 12,000,000 B.t.u./Hr. Other P.F.T. equipment at this plant includes spirally guided gas control holder lift for a 55' 6" dia. tank, floating cover position indicators, and a full complement of P.F.T. Gas Safety Equipment.

Sludge is pumped from the North Point Sewage Treatment Plant, over six miles away as well as from the Southeast Treatment Works, close by. The digested sludge from these ten digesters and from the Richmond-Sunset Treatment Plant is further processed by vacuum filtration and heat drying to provide a valuable supply of fertilizer for California growers. The engineering offices of Clyde C. Kennedy, San Francisco, were consulting engineers.

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Proceedings of Member Associations

OHIO SEWAGE AND INDUSTRIAL WASTES TREATMENT CONFERENCE

The 25th Annual Meeting of the Ohio Sewage and Industrial Wastes Treatment Conference was held in Columbus, Ohio, June 20–22, 1951. The registration numbered 221; 36 ladies also registered and enjoyed the activities arranged for them.

The morning of June 20 was devoted to registration and a Manufacturers' Forum. This forum stressed material of construction for corrosion resistance. Walter Sandel of Amercoat Corporation; Paul Harlamert, Johns-Manville; and Jack Cooper of Dubois-Cooper Associates discussed various aspects of the subject.

The afternoon session was opened with a resumé of "Federation Affairs," presented by Earnest Boyce, vice-president, Federation of Sewage and Industrial Wastes Associations. John D. Porterfield, director, Ohio Department of Health, then gave a resumé on the new anti-pollution law recently passed by the Ohio Legislature. This was followed by the business session of the Conference. The highlights of this portion of the program were a resumé of the past 24 Annual Conferences of the organization by C. D. McGuire, senior chemist, Columbus sewage treatment plant, and the first chairman; and the selection of Arthur H. Niles, engineer-superintendent, Toledo sewage treatment plant, to receive the Arthur Sidney Bedell Award. The afternoon program was completed by a report on the improvements at the Columbus sewage treatment plant by James H. Blodgett, superintendent.

The Thursday morning session opened with an Operators' Forum, at which the use of plug valves at sewage treatment plants and scum troubles in digesters were discussed. Following the forum, G. A. Hall, senior assistant engineer, Ohio Department of Health, presented a paper on "The Effects of Industrial Wastes on Sewage Treatment Processes." This paper was augmented by "Experiences at Fremont," by John Hess, superintendent of sewage treatment, Fremont; and "Experiences at Celina," by Carl Bauer, superintendent of sewage treatment, Celina, and Paul A. Uhlmann, consulting engineer, Columbus. Next was presented a paper on "Determination of Phenols by Amino-Antipyrine Method," by Michael Dannis, senior analyst, Research Division, Armco Steel Corporation, Middletown. A paper on "The Disposal of Radioactive Wastes," prepared by C. C. Ruchhoft and L. R. Setter, U.S.P.H.S. Environmental Health Center, Cincinnati, was presented by the latter.

Two sessions were scheduled for the afternoon, one dealing with industrial waste problems and the other with sewage treatment. In the industrial wastes section the session was opened with a panel entitled "Industry Organizes to Do the Job." Four representatives of industry presented a report on the activity of action committees formed under the guidance of the Ohio River Valley Water Sanitation Commission. E. J. Cleary, executive director and chief engineer of the Commission, was the moderator. On the panel were H. S. Kline, Frigidaire Division, General Motors Corporation, representing the metal finishing industry; Walker Penfield, Pennsylvania Salt Manufacturing Company, representing the chemical salts industry;

(*Continued on page 444a*)

Mobile's City Hall, a fine example of French colonial architecture, as it looked 100 years ago

The City of Mobile in Alabama has been under four flags since it was founded in 1702 as the capitol of the French province of Louisiana. As one of our older cities, Mobile quite naturally has cast iron water and gas mains in service that were laid well over a century ago. Thanks to the shock-strength, crushing-strength and beam-strength of cast iron pipe, these old mains continue to cope with stresses undreamed-of when they were installed. And because of these strength-factors of long life cast iron water and gas mains, laid over a century ago, are still serving in the streets of more than 30 American cities. United States Pipe and Foundry Company, General Offices, Burlington, N. J. Plants and Sales Offices Throughout the U. S. A.

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Frank Shipman, Brown-Fourman Distilleries, representing the distilling industry; and Grant A. Pettit, Armco Steel Corporation, representing the steel industry. This discussion was followed by three papers dealing with the program of milk wastes, as follows:

"Industrial Waste Problems in the Dairy Industry," by Frank J. McKee, sanitary engineer, Kraft Foods, Inc.

"Treatment of Milk Wastes, by Trickling Filters," by John W. Rugaber, district engineer, Pet Milk Company.

"Treatment of Dairy Wastes in Activated Sludge Type Plants," by Paul M. Thayer, district manager, Chicago Pump Company.

The sewage treatment section featured the following papers:

"Concentration of Waste Activated Sludge," by John J. Wirts and Frank J. Ausflug, superintendent and operator, respectively, Cleveland Easterly sewage treatment plant.

"A New Superintendent Takes Over," by Robert Bowman, superintendent of water and sewage, Newark, Ohio.

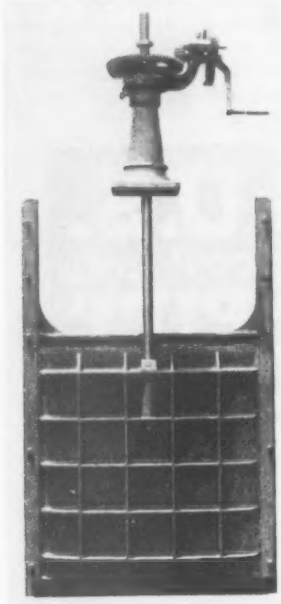
"Chemical Treatment of Sewage for Short Periods," by Ward E. Conrad, chief operator, Wooster sewage treatment plant.

"Report on Lake Erie Survey," by H. W. Streeter and H. P. Cowgill, consultant, Ohio Department of Natural Resources, and assistant engineer, Ohio Department of Health, respectively.

"Evaluation of Chemical Oxygen Demand Data," by Merrill L. Riehl, superintendent of water purification, Mahoning Valley Sanitary District, Youngstown.

The Annual Banquet was held on Thursday evening. The banquet was preceded by a cocktail party sponsored

(*Continued on page 446a*)



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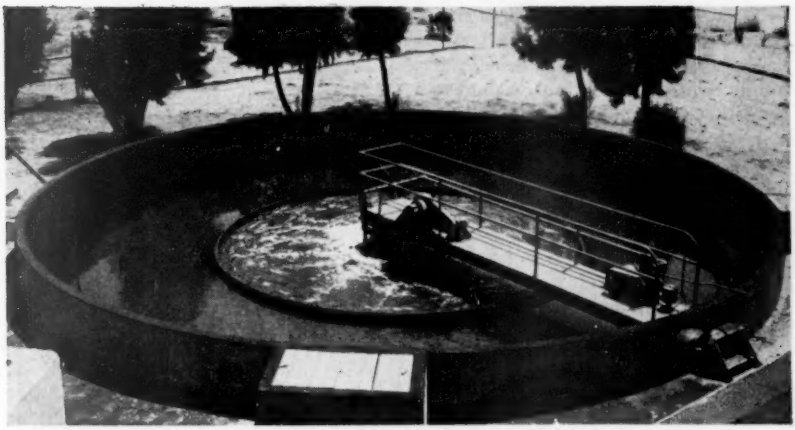

Palm Springs Process Oxidator. J. F. Davidson, Consulting Engineer. Karl P. Teget, Contractor.

PALM SPRINGS MODERNIZES SEWAGE TREATMENT WITH PROCESS OXIDATOR

Original One Tank Aeration, Coagulation and Sedimentation Unit with Sludge Recirculation as Pioneered by Process Engineers Again Proves Successful

California's famous desert resort, Palm Springs, used a conventional 40' Clarifier to serve the needs of a population that varies from less than 5,000 in the summer to more than 25,000 during the winter season. The low flow and high temperature of the sewage in the summer produced an odor which was objectionable to the nearby residents, while the increasingly heavy winter flow proved the clarifier inadequate. To remedy this condition, the Sanitary District of Palm Springs, upon the recommendation of its consulting engineer, chose to convert the clarifier to a Process Oxidator.

Since installation, this combination one tank aeration, coagulation and sedimentation unit with sludge recirculation, pioneered and developed by Process Engineers Incorporated, has eliminated the objectionable odor and greatly improved the clarification. (Note aeration in lower photo.)

This conversion, which has resulted in an intermediate treatment at approximately the cost of primary treatment, is the first step in a contemplated expansion program that will eventually include a biological filter and a secondary Oxidator for complete treatment. Having converted the conventional clarifier into an Oxidator, the BOD loading to the proposed biological filter will be greatly reduced, thereby reducing the size and cost of the biological filter.

The conventional clarifier was first installed during World War II when shortages of materials necessitated the use of wooden walkways, wooden weirs, wooden scum baffles, etc. At the time of conversion to an Oxidator, all wooden parts were replaced with steel.

The Palm Springs installation is another example of the flexibility and efficiency of the Process Oxidator. For information concerning this proven method of sewage treatment, either for improved clarification in existing clarifiers or for new installations, communicate with Process Engineers Incorporated, 6381 Hollywood Blvd., Los Angeles 28, California, or 212 Sutter Street, San Francisco 3, California. Eastern and Middle-Western representative: Pacific Flush Tank Company, 4241 Ravenswood Avenue, Chicago 13, Ill.

Close view of Oxidator showing Aeration. Patented and patent pending.

by the Water and Sewage Works Manufacturers' Association. The after dinner speaker was Harold K. Schellenger, public relations consultant, who gave a very interesting talk on public relations as related to the sewage and industrial wastes treatment field. This was followed by an enjoyable hour of magic tricks by Walter Simpson, Cambridge, Ohio.

The dual sessions were carried over into part of the Friday morning program. In the industrial wastes section the following papers were heard:

"Waste Elimination and Good Housekeeping in a Small Tomato Cannery," by George S. Davidson, general manager, Foster Canning Co., Napoleon.

"Waste Treatment and Disposal for Large Canneries Having Seasonal Operations," by C. W. Templeton, asst. chief engineer, Stokely Foods, Inc.

"Waste Treatment Trends in Large Canning Plants," by R. J. Dougherty, engineer, H. J. Heinz Company.

In the sewage treatment section the following papers were presented:

"Report on Short School for Water and Sewage Treatment Plant Operators," by W. D. Sheets, supervisor of school and assistant engineer, Ohio Department of Health.

"Formulation and Enforcement of Regulatory Sewer Ordinances," by A. D. Caster, senior engineer, Sewage Disposal Division, City of Cincinnati.

Movie: "Sewage Treatment Processes"—a film made principally in Ohio by U.S.P.H.S.

The following papers were heard at a combined session:

"Procedures for Sampling and Measuring Industrial Wastes," by H. H. Black, senior sanitary engineer, Industrial Wastes Section, Environmental Health Center, U.S.P.H.S., Cincinnati.

"Sedimentation and Flocculation Equipment for Treatment of Industrial

(*Continued on page 448a*)

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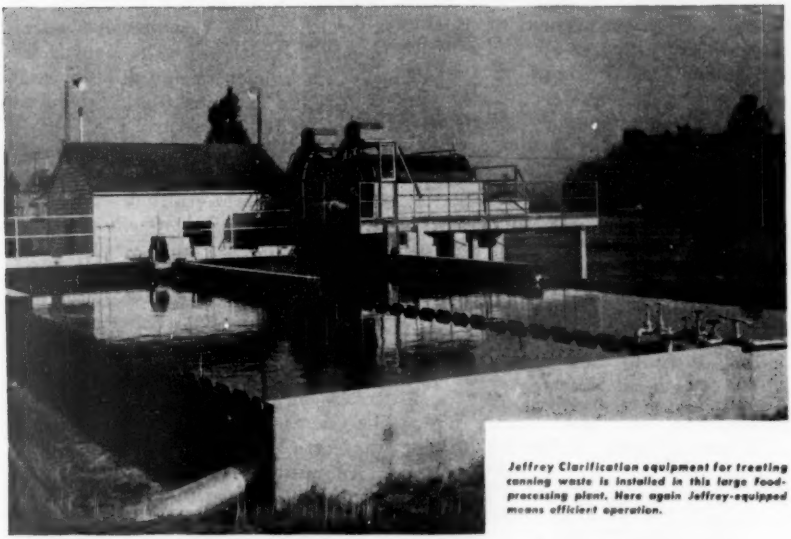

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Wastes," by J. H. Bass and R. R. Evans, field engineer and project engineer, respectively, Infilco, Inc.

"Evaluation of Methods for Cyanide Determinations," by E. G. Will, chief chemist, Ohio Department of Health Laboratory.

"Design and Operation of a Plant for Treating Plating Wastes," by C. M. Gard, consulting engineer, A. E. Stilson & Associates, Columbus; C. A. Snavely, assistant supervisor, Research Division, Battelle Memorial Institute, Columbus; and D. J. Lemon, chemical engineer, Ranco, Inc., Delaware.

The afternoon of June 22 was devoted to field trips. Arrangements were made for visits to the Columbus sewage treatment plant, Battelle Memorial Institute, and Ternstedt Division of General Motors.

Officers elected for 1951–52 were:

Chairman: Walter E. Gerdel, Cleveland.
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CANADIAN INSTITUTE ON SEWAGE AND SANITATION

The 18th Annual Convention of the Canadian Institute on Sewage and Sanitation was held at the Royal York Hotel, Toronto, Ont., on August 20–22, 1951. The registration of 359 set a new all-time record; the previous high had been 340, established in 1947.

After a pre-convention social evening on August 19, the sessions opened on August 20 with a guided discussion on "Problems with Tree Roots in Sewers." This was followed by the Convention Luncheon and papers on "An Analysis of Sewer Design Practice in Canada," by W. C. Miller, city engineer, St. Thomas, Ont., and "Municipal Finance in a Defense Economy," by G. A. Lascelles, Commissioner of Finance, Toronto, Ont. The Monday session concluded with a guided discussion on "Septic Tank Installations for Residences and Institutions."

The morning of August 21 was devoted to the Annual Business Meeting of the Institute; a paper describing the Etobicoke sewage treatment plant, by F. McArthur, engineer, Etobicoke Township; and a guided discussion on "Sewer and Water Crossings for Rivers and Ravines." The Past President's Luncheon was followed by an inspection trip of the Etobicoke sewage treatment plant, the North York and York Townships refuse incinerator, and the North York sewage treatment plant.

The Annual Banquet was held the evening of August 21, with a program of entertainment provided by the Canadian Sanitation Equipment Association.

The technical session on August 22 was devoted to a paper on "The Control of Oil Pollution in All Stages," by A. D. McRae, Imperial Oil Ltd., Sarnia, Ont., and a guided discussion on "Cost Estimating Under Present Conditions."

A "Club Room," courtesy of the Canadian Sanitation Equipment Association, was open at various times throughout the meeting when technical sessions were not in session.

Officers elected to serve during 1951–52 were:

President: C. S. Anderson, South Porcupine, Ont.
Vice-President: G. H. Baker, North York, Ont.
Secretary-Treasurer: A. E. Berry, Toronto, Ont.

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Rubber Pipe Gaskets—Engineering specifications for use of Hexseal rubber gaskets with reinforced concrete sewer pipe are contained in a new brochure (Specification 10) covering type, dimensions, and design of pipe; curing instructions; and installation.—Universal Concrete Pipe Co., 297 South High St., Columbus, Ohio.

Pipe Couplings and Sleeves—A new 34-page catalog (No. 513) gives a wealth of information on pipe clamps, sleeves, couplings, and expansion joints, as well as suggestions on time- and money-saving suggestions for repairing pipe leaks and breaks.—Dresser Manufacturing Division, Bradford, Pa.

Nozzles, Welding Necks, and Flanges—Besides describing and illustrating nozzles, necks, and flanges for pressure vessels and piping, Catalog 501 also includes data covering standards of the Tubular Equipment Manufacturers Association and a 44-page section on "Modern Flange Design." The latter, also available as a separate publication (Bulletin 502), contains much valuable handbook information and several time-saving design charts and sample calculation sheets.—Taylor Forge & Pipe Works, P. O. Box 485, Chicago 90, Ill.

Block Insulation—Properties and service recommendations for Superex high-temperature insulation, including conductivity and heat loss graphs and thickness tables, are given in a new 4-page folder.—Johns-Manville, 22 East 40th St., New York 16, N. Y.

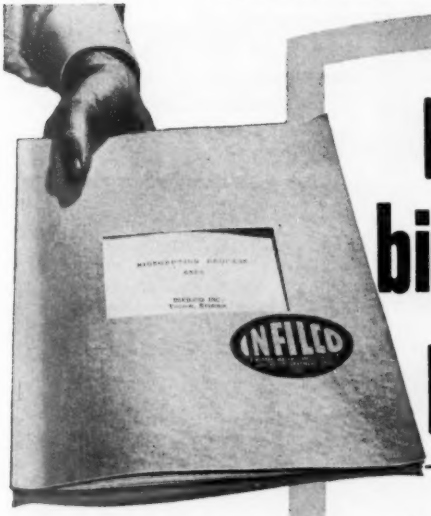
Centrifugal Pumps—Bulletin W-313-B1B describes Mixflo pumps in 20-in. to 84-in. sizes and capacities from 8,000 to 225,000 g.p.m. at heads of 5 to 50 ft. Dimensions for planning pump layouts are given.—Worthington Pump and Machinery Corp., Harrison, N. J.

Sequence Signal Transmission—A new 4-page bulletin (Sup. Bull. No. 230-H10) illustrates the equipment of Chronoflow sequence transmission and explains how the system provides for transmitting multiple functions over a single pair of wires. Also contained is an explanation of how the system can be extended to transmission in two directions, permitting of remote control and warning alarms.—Builders-Providence, Inc., 345 Harris Ave., Providence 1, R. I.

Filter Underdrains—Bulletin UF-3 describes and illustrates Unifilter block underdrains for trickling filters. Included are engineering data and specifications for regular and special shapes, filter wall tile, and filter design and construction.—National Fireproofing Corp., 327 Fifth Ave., Pittsburgh 22, Pa.

Recording Meters—Features of a recently announced new line of recording voltmeters and ammeters are described in a new 28-page bulletin (No. E1111). Complete information is given on ranges, uses, and specifications of the various models for permanent installation and portable use.—The Bristol Co., Waterbury 20, Conn.

Leak Detector—A new and effective method of detecting leaks of liquids in vessels of all kinds and sizes depends on the phenomenon of fluorescence generated by a soluble fluorescing agent and a new blacklight lamp. The new lamp is completely portable and battery operated.—Geo. W. Gates & Co., Inc., Franklin Square, L. I., N. Y.



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Max.	98.7	97.7
Min.	89.6	90.6
Avg.	93.6	94.0
Max.	97.7	97.5
Min.	88.0	88.9
Avg.	95.6	96.0
Max.	98.8	99.1
Min.	93.6	92.4

1 B.O.D. % Reduction

2 SUSPENDED SOLIDS % Reduction

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3 RETENTION

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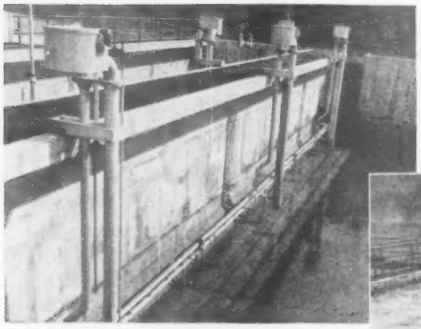
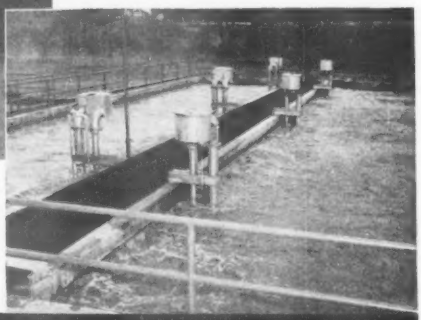


DIFFUSAIR installation at Kirkwood, Mo. Impingement nozzles and bowls are on headers at lower left side of tank.



DIFFUSAIR at Kirkwood, Mo. in operation showing effective "spiral flow" agitation and aeration of tank liquor.

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The principle involved is the combining of a low velocity jet of tank liquor and large air bubbles within an open bowl-like impinger. The cavitation effect thus produced divides the air into smaller bubbles than those diffused from a No. 30 permeability porous tube. The amount and pressure of the impingement water is very low when compared with the progressive back pressuring of the blower that persists when other types of air diffusers are employed.

The impingement liquor is derived directly from the aeration tank mixed liquor, and is lifted with a simple air-lift unit from which 1/3 of the fine air is re-entrained and utilized for oxygenation.

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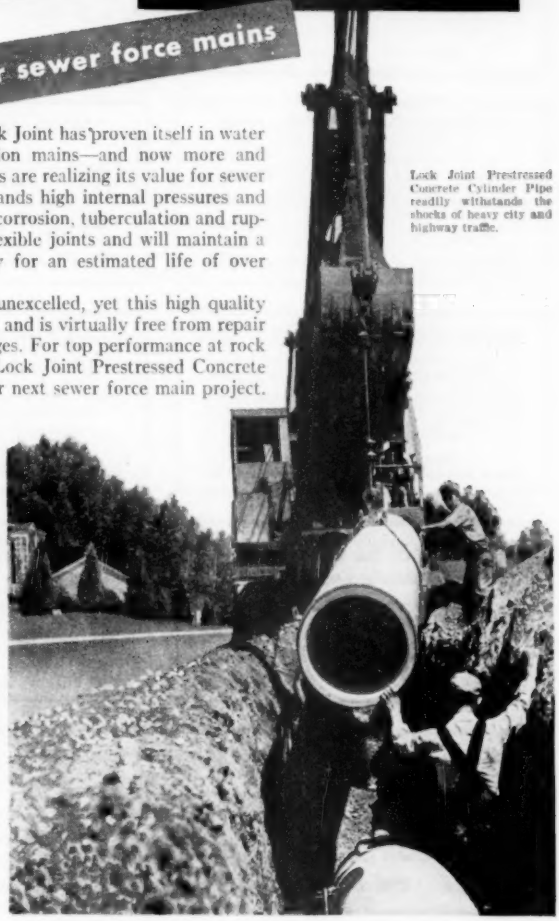

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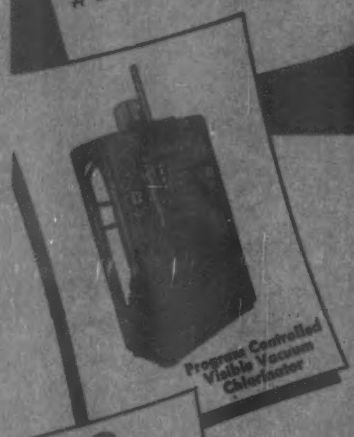

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